Jurnal Kejuruteraan 36(2) 2024: 471–480 https://doi.org/10.17576/jkukm-2024-36(2)-08

Optimization of Mechanical Properties Of Electrospun Epoxidized Natural Rubber/ Acrylonitrile-Butadiene-Styrene (ENR/ABS) Fibre For Membrane Application

Mahathir Mohamed^{a,c,} Abu Bakar Sulong^{a*}, Rosiah Rohani^{b,} Mohammed Iqbal Shueb^c, Mohd Sofian Alias^c & Mohd Hamzah Harun^c

^aDepartment of Mechanical & Manufacturing Engineering, Faculty of Engineering and Built Environment,

Universiti Kebangsaan Malaysia, Bangi, 43600 Selangor, Malaysia

^bDepartment of Chemical & Process Engineering, Faculty of Engineering and Built Environment,

Universiti Kebangsaan Malaysia, Bangi, 43600 Selangor, Malaysia

^cRadiation Processing Technology Division, Malaysian Nuclear Agency, Bangi, 43000 Kajang, Selangor, Malaysia

**Corresponding author: abubakar@ukm.edu.my*

Received 16 June 2023, Received in revised form 15 August 2023 Accepted 15 September 2023, Available online 30 March 2024

ABSTRACT

This study created membrane nanofibres using the electrospinning method and newly studied a mixture of ENR and ABS. The two-level complete factorial designs with centre points were used to characterise the functionality of the constructed membrane. The variables considered for experimental design were the polymer concentration, materials ratio (ENR concentration), applied voltage and distance between the needle tip and collector. According to the analysis of variance (ANOVA), the concentration of solution and distance were statistically significant parameters that affected the tensile properties of the ENR/ABS electrospun membrane. A mathematical model of the tensile property of polymer fibres was created using Response Surface Methodology (RSM). This model was built based on essential process factors. The mechanical properties of the electrospun ENR/ABS membrane compromised with 25wt% of solution concentration, 30% ratio of ENR, the voltage at 22.5kV and 15 cm of distance create an excellent tensile strength with desirability of 0.94. The influence of ENR on the morphology of ENR/ABS fibres was characterised by Scanning Electron Microscopy (SEM). The result showed beaded fibre and decreased fibres due to the low concentration of the solution and high ratio of ENR (50%). The contact angle measurements indicated that the electrospun fibre membrane was hydrophobic with a water contact angle of 136°. The addition of ENR showed a reduction in contact angle to 119°. The existence of ENR will change the features of the membrane, and investigations have demonstrated that RSM has been efficiently developed to acquire the interaction effects of processing parameters.

Keywords: Response surface methodology (RSM); Epoxidized natural rubber (ENR), electrospinning, hydrophilicity

INTRODUCTION

Electrospinning is a simple and versatile technique to produce ultrafine fibre in various materials, such as polymers, inorganic materials, and hybrid materials. This method makes it simple to create nanofibrous mats with desirable properties, including high functionality, high surface area to volume ratio, high porosity, and uniform shape (Ghosh et al. 2021; Ibrahim and Klingner 2020). In electrospinning, a polymer solution is subjected to a high voltage, which causes charged jets of solution to be driven through a spinneret and eventually into a grounded target, which acts as a collector (Elkasaby et al. 2018; Bolong et al. 2021).

In detail, a high voltage is applied to a polymer solution to produce an electrically charged jet. The solution is transformed into vapour before it reaches the collector and is then gathered as nanofibers. One electrode is connected to a metallic needle attached to a syringe, while the other is connected to the collecting region. The surface liquid becomes electrically charged as the electrostatic field intensity increases, and the repulsion between these charges produces shear forces. Because these pressures are opposite to surface tension, the initial hemispherical drop gradually elongates and transforms. When the potential strength of the electric field is raised, the hemispherical coat of the membrane at the tip of the syringe expands to form a conelike shape known as the Taylor cone.

Furthermore, by increasing the electric field, a threshold value is reached when the repulsive electrostatic force overcomes the surface tension and the charged jet of the solution is discharged from the Taylor cone's tip. The imbalance and extension process of the elongated polymer fluid is altered, allowing the jet to grow very long and thin. The solvent dissipates, leaving charged polymeric fibres randomly deposited onto the collector to form a nonwoven fibrous mat. Depending on polymer solution properties and processing parameters, the fibres can range from tens of nanometers to hundreds of micrometres. Fibre forming and spray forming are two possible outcomes of a solution jet travel (Wang et al. 2016; Angammana et al. 2011; Sarbatly et al. 2016).

The use of membrane technology is becoming more popular in the water industry due to its ability to disinfect water without chemicals and prevent the creation of harmful disinfection by-products. The membrane has evolved as a preferred filter medium for water treatment systems due to its benefits, such as minimal operation space, high filtration efficiency, and direct operational management (Jalloul et al. 2020; Wan At et al. 2019).

Electrospun nanofibres have several advantages over conventional fibres, such as a high surface area to volume ratio, an adaptable surface morphology, greater selectivity, customizable porosity, enhanced mechanical performance, and cost-effective operation (Roche and Yalcinkaya 2019). So, it has become an area of interest for many industries, including food packaging, tissue engineering, drug delivery, wound dressing, protective clothing, insulating materials, high-efficiency filtration, sensor and energy conversion (Baji et al. 2020; Khulbe and Matsuura 2019; Iregui et al. 2019). Due to their industrial viability, simplicity of operation, and high separation efficiency, electrospun membranes are frequently used for water purification. Depending on pore size and structure, ultrafiltration membranes have garnered considerable interest for the removal of proteins, bacteria, viruses, silica, pigments, polysaccharides, colloids, and emulsified oils from feed products, as well as for the purification of pharmaceutical products (Nikmaram et al. 2017).

| TADLE 1 | T1 / · | · · · | 1 . | 1 |
|----------|--------------|------------|--------------|-------------------|
| IABLE I. | Electrospinn | ing of cis | polvisoprene | research progress |
| | | 0 | | |

| | Materials System | Conditions | Tensile | Fibre diameter | References |
|---|---|--|---------|-----------------|--|
| 1 | 8 % w/v NR in toluene blend with 8 % w/v PCL in chloroform. 0, 2, 15, 30, and 50 % (v/v) NR/PCL. | Voltage: 20kV Distance: 12 cm Fixed grounded metal collector. | 5.1 MPa | 210 nm | (Maria et al. 2013) |
| 2 | 15 % w/v concentration of PLA/ENR blend dissolving in chloroform. | Voltage: 17.5kV Distance: 12 cm Flow rate: 0.5 ml/h | Nil | 858 - 1889 nm | (Cosme et al. 2016) |
| 3 | 16 % w/v Liquid epoxidized natural rubber (LENR)/PVC blend, dissolving in THF. | Voltage: 16 kV Distance: 15 cm | Nil | 3-5 μm | (Othman et al. 2013) |
| 4 | Natural rubber (NR)/ABS blend dissolving in THF. 15% w/v solution concentration. | Voltage: 15 kV Distance:15-25 cm Rotating circular at 1000 rpm | Nil | 5.78 – 22.96 µm | (Sithornkul and Threepopnatkul 2009) |
| 5 | 1–6 wt% of cis-1,4 polyisoprene dissolved in chloroform and dichloromethane. | Voltage: 11-15 kV Distance: 10-15 cm Rotating collector. | Nil | 20-60µm | (Hao et al. 2010) |

Many studies focus on polymer blends with natural rubber to improve their morphology and mechanical properties, such as PVC/ENR, ABS/NR, PVDF/ENR, PCL/ NR and many more (Othman et al. 2013; Sithornkul and Threepopnatkul 2009; Salaeh et al. 2014; Maria et al. 2013). The advantage of using ENR instead of natural rubber is the occurrence of an epoxy ring in the structure, which aims to enhance the properties and miscibility of polar polymers. Epoxidized natural rubber (50 mol%) epoxidation: ENR-50) has attracted great interest in polymer blend and composite research due to the epoxide group of ENR that can be used to enhance the system's compatibility. Interaction between the epoxide group of ENR and the polar group of the polymer is possible. On the other hand, using rubber as a secondary polymer can improve thermoplastic blend toughness. During stress testing, the rubber behaves as a stress concentrator, increasing the fracture energy absorption of brittle polymers and significantly improving the material's toughness. Instead of low-cost materials and one of the renewable resources, the combination of Natural rubber (NR) and epoxidized natural rubber (ENR) with other polymers would exhibit combined unique properties of the blends like toughness, flexibility, biocompatibility and biodegradability (Kargarzadeh et al. 2014).

In the current research, electrospun materials combining synthetic polymers with epoxidised natural rubber to improve their properties are rare, and only a few researchers have studied these mixtures. Table 1 demonstrates some advancements in natural rubber electrospinning research. In this work, copolymers of Acrylonitrile-butadiene-styrene (ABS) and low molecular weight of ENR (LENR) were blended until homogenous before being prepared using an electrospinning technique. ABS polymer is utilised extensively in the industry due to its excellent physical and mechanical properties, such as impact resistance and tensile strength. ABS also improves low-temperature durability, physical ageing, thickness and notch radius sensitivity, and, most importantly, processability. In the meantime, adding LENR would improve properties such as hydrophilicity, oil resistance, and thermal stability. Additionally, it would decrease surface tension, promote compatibility, and enhance the material's properties (Wu et al. 1994).

Response surface methodology (RSM) is a combination of mathematical and statistical techniques beneficial for modelling and optimising the effects of multiple independent variables on a performance measure or quality characteristic of a product or process (response) (Elkasaby et al. 2018). The Design of Experiment (DOE) method determines the relationship between the factors that impact the electrospinning process. This DOE was based on the twolevel (2k) with full factorial design. The 2-level full factorial design is particularly useful in the early phases of an investigation when the number of factors is less than or equal to four. This technique utilises statistical analysis of variance (ANOVA) and coefficients of determination (R2) to determine the influence of process parameters on responses. RSM provides a summary of the process parameters and their interrelationships (Enis et al. 2017; Ganj et al. 2019). A combination of ABS and low molecular weight ENR would have an essential impact on the tensile properties of electrospun non-woven membranes.

METHODOLOGY

MATERIALS

In this research, industrial grade Toyolac ABS (Acrylonitrile Butadiene Styrene) was purchased from Toray Industries.

473

Epoxidized natural rubber (ENR, 50 mol % conversion) was used as the modifier supplied by Guthrie Malaysia. Degradation of ENR via UV irradiation was done to form low molecular weight liquid epoxidized natural Rubber (LENR). Acetone as solvent was purchased from Sigma Aldrich Chemical Co.

PREPARATION OF ABS/LENR SOLUTION

ABS and LENR were dissolved in acetone at room temperature. The solutions were combined and vigorously agitated for five hours. The needle's inner diameter was 1.2 millimetres, and the solutions were put into a syringe with a capacity of 20 millilitres. The electrospun membrane's structural properties and morphological changes can be monitored using optical contact angle and scanning electron microscopy (SEM).

ELECTROSPINNING PROCESS

Synthesis of electrospun ABS nanofibre membranes was made using an electrospinning apparatus. The electrospinning apparatus, consisted of a highvoltage source, collector and syringe pump. The precursor solutions were put in a 20 mL syringe with a needle diameter of 1.2 mm. The sample in the syringe was installed on the syringe pump, which pushed the solution out of the syringe needle with a constant flow rate. The needle was connected to a high voltage (HV) source while the collector was grounded. The fabricated nanofibres were deposited on the aluminium foil wrapping the plate collector. The processing parameters during the electrospinning process were as follows: high voltage (15-30 kV), needle tip to collector distance (5-15 cm) and solution flow rate of 4 mL per hour.

FULL FACTORIAL DESIGN OF EXPERIMENT METHOD

Full factorials can be used to estimate the effects of all interactions. Analysis of variance (ANOVA) is a reliable and statistically significant method for determining the hypothesis on sample testing. ANOVA was utilized to gain significant parameters, thus affecting the quality of electrospun fibres and eventually attaining the optimum condition. The tensile properties of electrospun membrane fibre were carried out using a 2-level full factorial design and a total of 19 experimental runs. The parameters condition is shown in Table 2. These parameter settings were considered based on a preliminary study. Design Expert software version 10 (US, Stat-Ease Inc.) was used as a tool for data statistical analysis.

TABLE 2. List of parametres and their levels

| Parameter (unit) | Symbol | Level 1 | Level 2 | Level 3 |
|------------------------|--------|---------|---------|---------|
| Concentration (%) | А | 15 | 20 | 25 |
| Ratio (%) (ENR:ABS) | В | 10 | 30 | 50 |
| Voltage (kV) | С | 15 | 22.5 | 30 |
| Distance (cm) | D | 5 | 10 | 15 |

TENSILE TEST

The mechanical strength of electrospun fibre membranes was measured using the INSTRON Universal Tester 4381L machine. A sample of 10 mm in length and 5 mm in width was used for each measurement. All the tensile tests were carried out on dry membranes with a 0.6 mm/ min stretching rate at room temperature. Each test sample was secured between two cardboard frames of a sample holder before being clamped for testing. The sample holder was then cut at every side using scissors.

SCANNING ELECTRON MICROSCOPE (SEM)

Scanning Electron Microscope (SEM) model FEI Quanta 400 with 500x magnification was used to get information on fibre membrane morphology. Studies were performed in high vacuum mode at a voltage of 20 kV. Gold-coated samples were prepared in a vacuum (0.1–0.5 torr) with an inert gas (Argon) using the SC 50 sputter coater model. SEM observations were conducted on samples taken from the top of the fibre membrane.

OPTICAL CONTACT ANGLE

The water contact angle of the membranes has been measured at room temperature using Drop Shape Analysis (OCA 20). A 5μ L water droplet was placed on a membrane surface using a micro syringe with motorized control. Imaging software was used to acquire an image of a water particle on a membrane's surface and to measure the surface water contact angle. The contact angle for each membrane form was measured at a number of random locations, and the reported value is the mean of these values.

RESULTS AND DISCUSSION

FULL FACTORIAL DESIGN

The experimental design was carried out using a twolevel full factorial. Table 3 displays the values of the various variables of the multiple solutions generated following the levels of the design of experiment (DoE). Table 3 shows the influence of the four factors of control variables on the measured response, and 19 experiments were performed. The tensile strength distribution shown in bar graphs. The highest tensile value is obtained at sample 4 (1.14MPa). Accordingly, this is because perfectly uniform, electrospun fibres with a sufficient solid content were formed.

Meanwhile, the sample in the middle has a relatively high tensile value (samples 6, 12, and 13). Lower concentrations, such as samples 1, 5, 7, 9, 11, 14, and 16, cause a considerable drop in tensile strength (0.354, 0.612, 0.632, 0.532, 0.586, 0.655, and 0.446 MPa). The concentration positively affected the tensile as expected and has been previously mentioned in other studies (Asvar et al. 2017). Commonly, when low-concentration, it will develop beads and will cause fibres to have fewer interactions. Beads are typically called "by-products" in electrospinning and are detrimental to the membrane. Previous research demonstrates that as the number of nanofibre beads increases, so does the number of weak places, resulting in an overall low tensile strength (Tarus et al. 2016). Research has found that the beaded filaments are the result of several factors, including the discharge instability of the sample solution, the net charge density during electrospinning, the solution viscosity, and the surface tension of the solution. Figure 1a(1-2) shows sample 16 at a low concentration with numerous beaded fibres and sample 13 at a high concentration without beaded fibres.

ANALYSIS OF EXPERIMENTAL DESIGN

The results shown in Table 4 were analysed using analysis of variance (ANOVA) based on the mean response values for each run and the analysis statistics. It indicates the influence of control variables on measured responses. Design expert software gives an analysis of the experiment datasheet of the response surface design. This study revealed that the model is relevant through the F and p values. A small p-value and a higher F imply that the related variables are significant. The p-values are indicators of statistical significance, and the electrospinning parameter significantly affects the average fibre diameter when the Pvalue is less than 0.05 at a 95% confidence interval (Lee et al. 2019).

The *p*-values (significance) of the models are 0.0163 < 0.05 and *F* value is 4.26 for tensile strength. Meanwhile other significant model terms are *A* (concentration, p=0.0113<0.05), *D* (distance, p=0.0140<0.05) and CD (interaction of voltage and distance, p = 0.0474 < 0.05). There is no significant interaction between ratio and distance (p = 0.0957 > 0.05),

ratio, voltage and distance (p = 0.1373 > 0.05). The *R*-squared (R^2) value for tensile strength is 0.7306 or 73% variability in response. Therefore, the predicted models accurately represent the observed values and adequately explain the response. The predicted R2 of 0.4967 is in reasonable agreement with the adjusted R2 of 0.5592 (the

difference is less than 0.2). The lack of fit calculation (p > 0.05) suggested that the model still fits the data. Adequate precision is a measurement of signal-to-noise ratio, and the value 8.948 is an adequate signal (greater than 4 is desirable) (Tavakolmoghadam et al. 2019).

| Sample No. | A | B (D) (i) D() | C | D | Response |
|------------|--------------------|------------------|----------------|----------------|---------------------|
| | (Concentration, %) | (Ratio, %) | (Voltage, KeV) | (Distance, cm) | (Tensile, MPa) |
| 1 | 15 | 50 | 30 | 5 | $0.354{\pm}0.032$ |
| 2 | 25 | 50 | 15 | 15 | 0.826 ± 0.065 |
| 3 | 25 | 10 | 15 | 5 | 0.694 ± 0.122 |
| 4 | 25 | 50 | 30 | 15 | $1.140{\pm}0.098$ |
| 5 | 15 | 10 | 15 | 15 | 0.612 ± 0.144 |
| 6 | 20 | 30 | 22.5 | 10 | $0.900{\pm}0.063$ |
| 7 | 15 | 50 | 15 | 15 | 0.632 ± 0.036 |
| 8 | 25 | 50 | 15 | 5 | 0.800 ± 0.046 |
| 9 | 15 | 50 | 15 | 5 | $0.532{\pm}0.032$ |
| 10 | 25 | 10 | 30 | 15 | $0.878 {\pm} 0.045$ |
| 11 | 15 | 10 | 15 | 5 | $0.586{\pm}0.057$ |
| 12 | 20 | 30 | 22.5 | 10 | 0.998 ± 0.173 |
| 13 | 20 | 30 | 22.5 | 10 | 1.010 ± 0.050 |
| 14 | 15 | 10 | 30 | 15 | $0.655 {\pm} 0.052$ |
| 15 | 25 | 10 | 15 | 15 | $0.733 {\pm} 0.027$ |
| 16 | 15 | 10 | 30 | 5 | 0.446 ± 0.027 |
| 17 | 15 | 50 | 30 | 15 | $0.987 {\pm} 0.054$ |
| 18 | 25 | 10 | 30 | 5 | $0.847 {\pm} 0.096$ |
| 19 | 25 | 50 | 30 | 5 | 0.579 ± 0.057 |

TABLE 3. Experimental result of tensile strength using 2 level full factorial design

Equation (1) have been developed to demonstrate the effects of parameters.

TABLE 4. ANOVA for tensile

| Source | Sum of Squares | df | Mean Square | F Value | p-value | |
|---|----------------|----|-------------|---------|----------|-----------------|
| Model | 0.58 | 7 | 0.083 | 4.26 | < 0.0163 | significant |
| A-Concentration | 0.18 | 1 | 0.18 | 9.24 | < 0.0113 | significant |
| B-Ratio | 9.950E-003 | 1 | 9.950E-003 | 0.51 | 0.4888 | not significant |
| C-voltage | 0.014 | 1 | 0.014 | 0.71 | 0.4159 | not significant |
| D-distance | 0.17 | 1 | 0.17 | 8.51 | < 0.014 | significant |
| BD | 0.064 | 1 | 0.064 | 3.32 | 0.0957 | not significant |
| CD | 0.097 | 1 | 0.097 | 4.98 | 0.0474 | significant |
| BCD | 0.05 | 1 | 0.05 | 2.57 | 0.1373 | not significant |
| Residual | 0.21 | 11 | 0.019 | | | |
| Lack of Fit | 0.21 | 9 | 0.023 | 6.29 | 0.1447 | not significant |
| Cor Total | 0.79 | 18 | | | | |
| R2=0.7306, Adjusted R2=0.5592, Predicted R2=0.4967, Adequate precision=8.948 | | | | | | |

$$\begin{aligned} & \text{Fensile} = 0.75 + 0.11^*\text{A} + 0.025^*\text{B} + 0.029^*\text{C} + \\ & 0.10^*\text{D} + 0.063^*\text{BD} + 0.078^*\text{CD} + 0.056^*\text{BCD} \end{aligned} \tag{1}$$

Figure 1(b-1) is a graphical analysis of the predicted versus actual plot. The actual distribution of tensile strength closely matches the predicted straight line. These results indicate that the chosen parameters have some effect on the response, so the model can be considered reliable and repeatable.

Figure 1(b-2) shows the pareto graph for each parameter that influences the tensile strength. Two parameters have a significant effect on the response of tensile, which are concentration (A) and distance (D). These two parameters give a positive effect of tensile value that was increased with increased concentration or distance. The interaction parameters that can be considered to provide some effect on the response are CD, BD and BCD.

Figure 1c shows three-dimensional response surface graphs created to analyze how the variables interact with each other and affect the response. The data we have collected shows that high levels of distance, concentration, ENR ratio, and voltage can lead to greater tensile strength. Figures 1(c-1) to (c-3) showed an increase in tensile yield when the variable was at maximum value. However, the tensile yield dropped when the variable was at the minimum value. Figure 1(c-1) depicts the influence of ENR ratio and distance on tensile strength. When we changed the ENR ratio, the distance of 5cm did not influence tensile strength. When the distance was increased to 15cm, it was discovered that the ENR significantly improved the tensile strength. Tensile strength increased because of the sufficient time to develop full micro-nanofibre and the formation of fewer bead fibres. To a long distance, the fibres were straight and cylindrical, indicating that the fibre was largely dry when it arrived at the collection. The distance between the needle tip and the collector influenced the deposition time, solvent evaporation rate, and instability interval (Subbiah et al. 2005).

Moreover, the impact of the ENR ratio at 50% ENR makes fibre formation in electrospinning difficult. One possible reason for this could be that rubbers have lower glass transition temperatures compared to room temperature. Additionally, their high elasticity might hinder the formation of fibres during electrospinning. In addition, the macromolecular chains of rubbers can move (i.e., rearrange their conformations) in electrospun fibres. This movement and relaxation occur when the stretching force is no longer present and is driven by increased entropy (Hu et al. 2012).

Figure 1(c-2) also demonstrates an influence on tensile strength. For a relatively short distance, the electric field got very strong, increasing the instability of the jet solution and allowing many jets to emerge from a single nozzle, producing bead fibres (Aliabadi 2017). Meanwhile, raising the voltage causes more charges to be generated in the

solution or at the tip of the needle, which is linked to a stronger electrical field (greater electrostatic forces), stretching the jets and forming a uniform or smooth fibre (Zhu et al. 2010). Increasing the distance between the collector and the needle tip and the voltage results in a suitable combination for forming solid micro-nanofibres. In addition, it will affect the duration of the solvent's evaporation, the electric field's strength, and the position of the electrical stress.

In Fig. 1(c-3), it is clear that a reduction in polymer concentration has an adverse effect on tensile strength, while an increase has a positive effect. This is because low concentration results in low viscosity, making it easier to form beads. Beads are a flaw that interferes with the performance of fibres. Meanwhile, increased concentration causes a rise in viscosity, leading to increased chain entanglement among the polymer chains. These chain entanglements can resist surface tension, resulting in increased fibre diameter, uniformity, and smooth fibre free of beads. When all other factors stay the same, an increase in solution concentration causes the viscoelastic force to increase gradually, stopping the electrostatic and columbic repulsion forces from stretching the fibres. (Tarus et al. 2016).

The optimisation of the responses of tensile strength is presented in Figures 1(d-1) to (d-5). The acceptable values of the desirability function (D) were close to one (100%). Desirability is merely a mathematical way to identify the optimal. A desirability function (D) assigns values between 0 and 1. Zero is an unacceptable reaction, whereas one is ideal ($0 \le D \le 1$) (Rabbi et al. 2012).

In this work, the mechanical characteristics of the electrospun ENR/ABS membrane compromised with 25 wt% of solution concentration, 30% ratio of ENR, the voltage at 22.5kV and 15 cm of distance provide a satisfactory tensile strength with the desirability of D=0.94. These values of the independent variables showed an increase in responses of tensile strength 0.96 MPa, respectively. Using numerical optimisation, we may pick a suitable value for each input factor and response. Different input optimisation techniques, including range, maximum, minimum, target and none, may be chosen to generate an optimum output value. This study put variables like ENR ratio and voltage at a specific target (22.5kV and 30% of ENR) because the fibre formation at this target is uniform and has a smooth surface without beads.

Figure 1f shows the stress–strain behaviour of the tensile testing of specimens made from the optimum and lowest tensile strength value of ABS/ENR electrospun fibre membrane. Sample 13 (S13) exhibits higher tensile strength and strain (σ_{max} : 1.4 MPa, ϵ : 20%) value compared to sample 16 (S16) (σ_{max} : 0.4 MPa, ϵ : 14%) due to bead formation. Beads act as defects and have fewer fibre-to-fibre interactions. As the concentration increase, smoother

476

fibres with improved diameter uniformity are formed, increasing fibre cohesion points, thus increasing the tensile strength.

SCANNING ELECTRON MICROSCOPY (SEM)

In Fig. 1(g-1) and 1(g-2), SEM images show that pure ABS and 70:30 (ABS: ENR) generates uniform fibres with no bead formation. Nevertheless, raising the ENR concentration to a 50:50 ratio (Fig. 1(g-3)) produces both the electrospun bead structure and uniform electrospun fibres. This is because the fibres fail to completely dry out before reaching the collector, mainly when the ENR level is high. The formation of beads demonstrates insufficient chain entanglements. The concentration will be slightly disrupted by an increase in the ENR ratio in the solution, and the fibre morphology changes. The competition between surface tension and viscosity is due to the solution's concentration (Zheng et al. 2014). The viscosity of the solution is related to the degree of the entanglement of polymer chain molecules within the solution. During the electrospinning process, a solution with a low viscosity will have a low viscoelastic force, which means that it will not be able to meet the electrostatic and columbic repulsion forces that stretch the electrospinning jet. Because of this, the jet will undergo segmental fragmentation as a result (Sosiati et al. 2018). Under the influence of surface tension, a large number of free solvent molecules in a solution form spherical aggregates, resulting in the formation of beads.

The dependence of fibre diameter on solution composition is observed in higher magnification (Fig. 1(g-1a) to (g-3a)). We can see that (Fig. 1(g-1a) and (g-2a)) the fibres are beadles and finely electrospun fibrous structures with the appearance of continuous linear and random orientation. Electrospun ABS/ENR fibres had a slightly smaller diameter than pure ABS fibres. This was due to the polarity in the ENR backbone (for example, ENR-50 implies 50 mol% of epoxide concentration), which caused the diameter to be somewhat lower. As described in the literature, increasing the conductivity of the electrospinning solution decreases the fibre diameter (Abdollahi et al. 2019). The averages of fibre diameter were calculated using Image J software and show normal distribution. The morphology observed that the average size of fibres shifted toward smaller diameters as the ENR ratio increased (Figure (g-1b) to (g-3b)).

WATER CONTACT ANGLE

The water contact angle determines the surface hydrophilicity and wettability of the membrane. The lower water contact angle generally describes more excellent hydrophilicity; a higher contact angle is associated with lower membrane surface hydrophilicity (Zhang et al. 2015). Figures 1(e-1) to 1(e-4) show the water contact angle (WCA) of the electrospun fibre mats of the ABS and ENR/ABS with different mass ratios. WCA studies were done to determine how the amount of ENR affected the wettability of the membrane. ABS has a high hydrophobicity, so adding ENR has some effect on making it more water-friendly.

The Electrospun of pure ABS has a water contact angle of 136°, while the ABS/ENR electrospun membrane showed a slight WCA decrease with the addition of ENR at 10wt% (123°). By increasing the content of ENR in electrospun membranes to 30wt% and 50wt%, the water contact angle decreased to 122° and 119° due to hydroxyl groups and polarity in ENR (Shen et al. 2017). The changes in hydrophilicity predict compatibility between ABS and ENR, which is polar compounds with polar hydroxyl, epoxy, and carboxyl groups.

The contact angle distribution of the electrospun ABS/ENR membrane showed a trend of decreasing contact angle values which is the evidence that the wettability of the ABS/ENR membrane improves as ENR increases.

CONCLUSIONS

this study, the electrospinning technique In successfully prepared the electrospun ENR/ABS membrane. The interaction of low molecular weight LENR and ABS in spinning solutions affects the solution viscosity and spinning ability. The investigation of the electrospinning parameters, including solution concentration, ENR ratio, applied voltage and distance between tip to the collector concentration, showed some influence on the response of membrane tensile strength. The ANOVA result showed that this model is relevant when the *p*-value is <0.05. Meanwhile, factors A, D, and CD are statistically significant compared to other factors. Factor B, C, BD and BCD showed statistically insignificant. Nevertheless, it does not mean these factors are unimportant, but it implies a minor influence on fibre formation and response. Based on the results, the electrospinning process's optimal conditions were a solution concentration of 25%, ENR ratio of 30%, applied voltage of 22.5 kV and tip-collector distance of 15 cm. The SEM image showed that the ABS/ENR blending solution gives smooth and uniform micro-nanofibres and enhances desired membrane properties. The water contact angle result showed some improvement in hydrophilicity with ENR content and would contribute to developing a membrane for water treatment.



FIGURE 1. (a) SEM morphology of sample (a-1)16 and (a-2)13; (b-1) Predicted versus actual data for tensile strength; (b-2) Pareto chart of standardized effects; (c) Response surface for tensile properties of electrospun membrane, (c-1) Ratio and distance (c-2) Voltage and distance (c-3)
Concentration and distance; (d) Optimization using the desirability function; (f) Stress-strain curve of ABS/ENR electrospun fibres membrane; (g) Effect of ABS: Epoxidised Natural Rubber (ABS:ENR) ratio on micro-nanofibre morphology, 2000x magnification (g-1) 100:0 (g-2) 70:30 (g-3) 50:50, 5000x magnification (g-1a) 100:0 (g-2a) 70:30 (g-3a) 50:50 and Fibre Diameter (g-1b) 100:0 (g-2b) 70:30 (g-3b) 50:50; (e) Effect of Epoxidised Natural Rubber (ENR) content on water contact angle (WCA), (e-1) ABS, (e-2) 90:10, (e-3) 70:30, (e-4) 50:50 (ABS:ENR)

ACKNOWLEDGEMENT

The authors would like to thank the Malaysia Ministry of Higher Education for sponsoring this work through Grant No. GUP-2019-007.

DECLARATION OF COMPETING INTEREST

None

REFERENCES

- Abdollahi, Serveh, Morteza Ehsani, Jalil Morshedian, Hossein Ali Khonakdar. & Elham Aram. 2019. Application of Response Surface Methodology in Assessing the Effect of Electrospinning Parameters on the Morphology of Polyethylene Oxide/ Polyacrylonitrile Blend Nanofibres Containing Graphene Oxide. *Polymer Bulletin* 76 (4): 1755–73.
- Aliabadi, Majid. 2017. Effect of Electrospinning Parameters On The Air Filtration Performance Using Electrospun Polyamide-6 Nanofibres. *Chemical Industry and Chemical Engineering Quarterly* 23(4): 441–446.
- Abdollahi, Serveh, Morteza Ehsani, Jalil Morshedian, Hossein Ali Khonakdar. & Elham Aram. 2019. Application of Response Surface Methodology in Assessing the Effect of Electrospinning Parameters on the Morphology of Polyethylene Oxide/ Polyacrylonitrile Blend Nanofibres Containing Graphene Oxide. *Polymer Bulletin* 76 (4): 1755–73.
- Aliabadi, Majid. 2017. Effect of Electrospinning Parameters On The Air Filtration Performance Using Electrospun Polyamide-6 Nanofibres. *Chemical Industry and Chemical Engineering Quarterly* 23(4): 441–446.
- Angammana, Chitral J. & Shesha H. Jayaram. 2011. Analysis of the effects of solution conductivity on electrospinning process and fiber morphology. *IEEE Transactions on industry applications* 47(3): 1109– 1117.
- Asvar, Zahra, Esmaeil Mirzaei, Negar Azarpira, Bita Geramizadeh & Milad Fadaie. 2017. Evaluation of Electrospinning Parameters on the Tensile Strength and Suture Retention Strength of Polycaprolactone Nanofibrous Scaffolds through Surface Response Methodology. *Journal of the Mechanical Behavior* of Biomedical Materials 75 (March): 369–78.
- Baji, Avinash, Komal Agarwal & Sruthi Venugopal Oopath. 2020. Emerging Developments in the Use of Electrospun Fibres and Membranes for Protective. *Polymers* 12(2): 492.

- Bolong, Nurmin & Ismail Saad. 2022. Optimizing Fabrication of Electrospinning Nanofiber Membranes for Water Filtration using Response Surface Methodology. *Jurnal Kejuruteraan* 34, no. 5: 975–982.
- Cosme, Jaqueline G. L., Vanessa M. Silva, Regina R. C. Nunes & Paulo H. S. Picciani. 2016. Development of Biobased Poly(Lactic Acid)/Epoxidized Natural Rubber Blends Processed by Electrospinning: Morphological, Structural and Thermal Properties. *Materials Sciences and Applications* 07 (04): 210– 219.
- Elkasaby, Mohamed, Hussien A Hegab, Atef Mohany & Ghaus M Rizvi. 2018. Modeling and Optimization of Electrospinning of Polyvinyl Alcohol (PVA). *Advances in Polymer Technology* 37(6): 2114–2122.
- Enis, Ipek Y, Hande Sezgin & Telem G Sadikoglu. 2017. Full Factorial Experimental Design for Mechanical Properties of Electrospun Vascular Grafts. *Journal of Industrial Textiles* 47(6): 1378–1391.
- Ganj, Mohammad, Mahdieh Asadollahi & Seyyed Abbas Mousavi. 2019. Surface Modification of Polysulfone Ultrafiltration Membranes by Free Radical Graft Polymerization of Acrylic Acid Using Response Surface Methodology. *Journal of Polymer Research* 26: 1–19.
- Ghosh, Tanushree, Trisha Das & Roli Purwar. 2021. Review of Electrospun Hydrogel Nanofibre System: Synthesis, Properties and Applications. *Polymer Engineering and Science* 61 (7): 1887–1911.
- Hao, Xiufeng, Chenxi Bai, Yue Huang, Jifu Bi & Chunyu Zhang. n.d. 2010. Preparation of Cis
 -1 , 4-Polyisoprene Electrospun Microfibres. Macromolecular Materials and Engineering 295(4): 305–309
- Hu, Q, H Wu, L Zhang, H Fong & M Tian. 2012. Rubber Composite Fibres Containing Silver Nanoparticles Prepared by Electrospinning and In-Situ Chemical Crosslinking. *Express Polymer Letters* 6(4): 258– 265.
- Ibrahim, Hassan M. & Anke Klingner. 2020. A Review on Electrospun Polymeric Nanofibres: Production Parameters and Potential Applications. *Polymer Testing* 90: 106647.
- Iregui, Alvaro, Lourdes Irusta, Loli Martin & Alba González. 2019. Analysis of the Process Parameters for Obtaining a Stable Electrospun Process in Different Composition Epoxy/Poly ε-Caprolactone Blends with Shape Memory Properties. *Polymers* 11(3): 475.
- Jalloul, Ghadeer, M Hadi Hashem, Ali Reza, Mohammad N Ahmad & Belal J Abu. 2021. Unsupported Electrospun Membrane for Water Desalination Using Direct Contact Membrane Distillation. *Journal of Applied Polymer Science* 138(7): 49861.

Kargarzadeh, Hanieh, Ishak Ahmad, Ibrahim Abdullah, Raju Thomas, Alain Dufresne, Sabu Thomas, and Aziz Hassan. 2015. "Functionalized Liquid Natural Rubber and Liquid Epoxidized Natural Rubber : A Promising Green Toughening Agent for Polyester. Journal of applied polymer science 132(3).

- Khulbe, Kailash Chandra & Takeshi Matsuura. 2019. Art to Use Electrospun Nanofibres / Nanofibre Based Membrane in Waste Water Treatment, Chiral Separation and Desalination. *Journal of Membrane Science and Research* 5(2): 100–125.
- Lee, C., L. Hun, H. Yaakob, S. L. Wong, & B. J. Hichem. 2019. Optimization of ultrasound-assisted extraction of total flavonoids content from the white flowering variety of Melastoma Malabathricum. *Jurnal Kejuruteraan* 2: 91–102.
- Mahmoudi, Ebrahim, Law Yong Ng, Wei Lun Ang, Ying Tao Chung & Rosiah Rohani. 2019. Enhancing Morphology and Separation Performance of Polyamide 6, 6 Membranes By Minimal Incorporation of Silver Decorated Graphene Oxide Nanoparticles. *Scientific reports* 9(1): 1–16.
- Maria, Ligia, Manzine Costa, Luiz Henrique, and Capparelli Mattoso. 2013. Electrospinning of PCL
 / Natural Rubber Blends. *Journal of materials* science 48: 8501–8508.
- Nikmaram, Nooshin, Shahin Roohinejad, Sara Hashemi, Mohamed Koubaa, Francisco J. Barba, Alireza Abbaspourrad & Ralf Greiner. 2017. Emulsionbased systems for fabrication of electrospun nanofibers: Food, pharmaceutical and biomedical applications. *RSC advances* 7(46): 28951–28964.
- Othman, Muhammad Hariz, Mahathir Mohamed & Ibrahim Abdullah. 2013. Electrospinning of PVC with Natural Rubber. In *AIP conference Proceedings* 1571(1): 926–931.
- Rabbi, Amir, Komeil Nasouri, Hossein Bahrambeygi, Ahmad Mousavi Shoushtari & Mohammad Reza Babaei. 2012. RSM and ANN Approaches for Modeling and Optimizing of Electrospun Polyurethane Nanofibres Morphology. *Fibres and Polymers* 13(8): 1007–14.
- Roche, Remi & Fatma Yalcinkaya. 2019. Electrospun Polyacrylonitrile Nanofibrous Membranes for Point of Use Water and Air Cleaning. *ChemistryOpen* 8(1): 97–103.
 Salaeh, Subhan, Gisèle Boiteux, Olivier Gain,
- Salaeh, Subhan, Gisèle Boiteux, Olivier Gain, Philippe Cassagnau & Charoen Nakason. 2014. Dynamic Mechanical and Dielectric Properties of Poly(Vinylidene Fluoride) and Epoxidized Natural Rubber Blends. In Advanced Materials Research 844: 97–100.
- Sarbatly, Rosalam, Duduku Krishnaiah, and Zykamilia Kamin. 2016. A review of polymer nanofibres by electrospinning and their application in oil–water separation for cleaning up marine oil spills. *Marine pollution bulletin* 106(1-2): 8–16.
- Shen, Liguo, Shushu Feng, Jianxi Li, Jianrong Chen, Fengquan Li, Hongjun Lin & Genying Yu. 2017. Surface Modification of Polyvinylidene Fluoride (PVDF) Membrane via Radiation Grafting: Novel

Mechanisms Underlying the Interesting Enhanced Membrane Performance. *Scienti ic Reports* 7 (1): 1–13.

- Sithornkul, Sarawuth, and Poonsub Threepopnatkul. 2009. Morphology of Electrospun Natural Rubber with Acrylonitrile-Butadiene-Styrene. *Advanced Materials Research* 79–82: 1583–86.
- Sosiati, Harini, Alif Nur Widodo & Aris Widyo Nugroho. 2018. The Influence Of Aloe Vera Concentration On Morphology And Tensile Properties Of Electrospun Aloe Vera-PVA Nanofibre. Jurnal Sains Materi Indonesia 19 (4): 157–162.
- Subbiah, Thandavamoorthy, Gajanan S. Bhat, Richard W. Tock, Siva Parameswaran & Seshadri S. Ramkumar. 2005. Electrospinning of nanofibers. *Journal of applied polymer science* 96(2): 557–569.
- Tarus, Bethwel, Nermin Fadel, Affaf Al-Oufy, and Magdi El-Messiry. 2016. Effect of polymer concentration on the morphology and mechanical characteristics of electrospun cellulose acetate and poly (vinyl chloride) nanofiber mats. *Alexandria Engineering Journal* 55(3): 2975–2984.
- Tavakolmoghadam, Maryam, Amir Mokhtare, Fatemeh Rekabdar & Majid Esmaeili. 2019. A predictive model for tuning additives for the fabrication of porous polymeric membranes. *Materials Research Express* 7(1): 015312.
- Wan At, Wan Nur Syuhada, Ang Wei Lun & Abdul Wahab Mohammad. 2019. Role of graphene oxide in support layer modification of thin film composite (TFC) membrane for forward osmosis application." Jurnal Kejuruteraan 31(2): 327–334.
- Wang, Shan, Xinglei Zhao, Xia Yin, Jianyong Yu & Bin Ding. 2016. Electret polyvinylidene fluoride nanofibers hybridized by polytetrafluoroethylene nanoparticles for high-efficiency air filtration. ACS applied materials & interfaces 8(36): 23985–23994.
- Wu, Jiann-shing, Shu-chen Shen & Feng-chih Chang. 1994. Effect of rubber content in acrylonitrile– butadiene–styrene and additional rubber on the polymer blends of polycarbonate and acrylonitrile– butadiene–styrene. *Polymer journal* 26(1): 33–42.
- Zhang, Chunmei, Max R. Salick, Travis M. Cordie, Tom Ellingham, Yi Dan & Lih Sheng Turng. 2015.
 Incorporation of Poly(Ethylene Glycol) Grafted Cellulose Nanocrystals in Poly(Lactic Acid) Electrospun Nanocomposite Fibres as Potential Scaffolds for Bone Tissue Engineering. *Materials Science and Engineering C* 49: 463–471.
- Zheng, Jian-Yi, Ming-Feng Zhuang, Zhao-Jie Yu, Gao-Feng Zheng, Yang Zhao, Han Wang & Dao-Heng Sun. 2014. The effect of surfactants on the diameter and morphology of electrospun ultrafine nanofiber. *Journal of Nanomaterial* 8–8.
- Zhu, Jiahua, Suying Wei, Xuelong Chen, Amar B. Karki, Dan Rutman, David P. Young & Zhanhu Guo. 2010. Electrospun polyimide nanocomposite fibers reinforced with core- shell Fe-FeO nanoparticles. *The Journal of Physical Chemistry* C 114(19): 8844–8850.

480