

# Chemical Engineering & Process Techniques

#### Research Article

# Electrospinning of Sulfobetaine Methacrylate Nanofibers

# Erin Emerick<sup>1</sup>, Sheila Grant<sup>2</sup> and Matthew Bernards<sup>1,2\*</sup>

<sup>1</sup>Department of Chemical Engineering, University of Missouri, Columbia, USA

#### Abstract

Sulfobetaine methacrylate (SBMA) is a zwitterionic polymer that has previously been shown to have nonfouling properties. This resistance to nonspecific protein adsorption makes the polymer a candidate for filtration applications where irreversible fouling is problematic. In this work, the preparation of SBMA nanofibers by electrospinning was investigated. SBMA was polymerized to different molecular weights by controlling the polymerization reaction solution.

In order to electrospin SBMA nanofibers, polySBMA solutions were prepared by dissolving the polySBMA in aqueous NaCl. When all other electrospinning conditions were held constant, it was shown that as the molecular weight of the polySBMA was increased, there was a related increase in the resulting fiber diameter. This can be attributed to the interrelated effects of the polymer size and the viscosity of the electrospinning solution. The effect of the concentration of NaCl in the electrospinning solution was then probed for the smallest molecular weight polySBMA. A step change in fiber diameter was seen as the concentration of NaCl was increased from 0.25 M to 0.5 M. However, the middle two salt concentrations showed the most consistent fiber diameters over the range of salt concentrations investigated. Electrospun polySBMA materials represent a promising new approach for standalone filtration membranes.

#### Corresponding author

Matthew Bernards, Departments of Chemical Engineering and Biological Engineering, University of Missouri, Columbia, MO 65211, USA, Email: bernardsm@missouri.edu

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#### Keywords

- Sulfobetaine methacrylate
- Nonfouling; Electrospinning

#### **INTRODUCTION**

A common problem with membrane filtration systems is membrane fouling [1-3]. Fouling can quickly and significantly decrease the flux through a membrane, making it less efficient and reducing its operational lifespan. Membrane fouling is typically characterized as one of two types: reversible and irreversible [4]. Reversible fouling occurs when the particles that are being filtered out of the solution become entrapped within the natural pore structure of the membrane. This type of fouling is inevitable and it can usually be removed with a back flushing process. Irreversible fouling occurs when molecules like proteins adsorb onto the membrane surface. This irreversible fouling process can lead to the blockage of the underlying pore structure, leading to a loss of flux. However, it also reduces the efficiency of the back flushing process leading to a further reduction in the performance as a result of reversible fouling. Irreversible fouling cannot be easily removed and therefore it is the major factor dictating the overall performance and lifetime of the membrane [5,6].

One approach for limiting the amount of irreversible fouling that occurs in a filter is to integrate nonfouling functional groups. By manufacturing a membrane out of a polymer that is naturally protein resistant or by modifying the surface chemistry of the material to incorporate nonfouling functional groups, the amount

of nonspecific protein adsorption can be drastically reduced [1,7-9]. As such, many investigators have investigated polymers that are considered to be nonfouling. One of the most common groups of nonfouling polymers is the poly (ethylene glycol) (PEG) and oligo (ethylene glycol) (OEG) family [10-12]. While this family of materials has been widely investigated and has led to improvements in membrane performance, they have not yet been able to completely eliminate irreversible fouling from membrane materials.

Another class of materials that have been widely investigated for their nonfouling properties are zwitterionic polymers [13-18]. Zwitterionic polymers are distinguished by the presence of both a negatively and positively charged moiety within their pendant groups. This characteristic has been shown to contribute to protein resistance [19]. A number of zwitterionic polymers have been investigated for their nonfouling properties including phosphorylcholine [13,20], carboxybetaine [16,17,21], and sulfobetaine [15,22,23]. The focus of this investigation is on poly (sulfobetaine methacrylate) (polySBMA), whose monomer structure is shown in Figure 1. PolySBMA has been shown to have excellent resistance to nonspecific protein adsorption even in complex medium [14] and it was seen to be more effective at preventing bacteria adhesion than many other nonfouling materials [24]. It has also shown promise for reducing irreversible fouling when surface grafted to an existing poly(vinylidene

<sup>&</sup>lt;sup>2</sup>Department of Biological Engineering, University of Missouri, Columbia, USA



fluoride) (PVDF) membrane [7,25]. These studies suggest that polySBMA has good potential for use as a stand-alone polymer membrane material.

Currently there are many different techniques being used to make filtration membranes and to change their surface properties to provide improved resistance to irreversible fouling. Membranes can be created from a polymer film using phase inversion on a substrate [6] or by weaving together fibers of a desired material [26]. Additionally, it is possible to modify the surface chemistry of an existing membrane material through surface grafting or other procedures [7,25]. A relatively new technique being used to create filtration membranes is electrospinning [27-29]. In electrospinning, fibers are formed by feeding a polymer solution through a metal capillary while applying voltage to the tip. This creates an electrostatic repulsion between the solution and the tip, leading to the ejection of a jet of micro-or nanoscale polymer fibers.

There are many benefits to using the electrospinning technique to make filtration membranes [30]. Electrospinning is a simple process, and the instrumentation is easy to set up and scale up for large-scale manufacturing. It is also a very inexpensive technique and easy to operate. An important aspect of this technique is its ability to be fine-tuned to control various fiber characteristics [31,32]. There are many aspects of the process that can be adjusted in order to affect fiber diameter, fiber alignment and configuration, pore size, etc. Research has shown that electrospinning can create membranes with more uniform fibers than some other techniques currently being used [33]. Furthermore, it is possible to create a nonfouling polymer filtration membrane directly by electrospinning. This will eliminate the need for additional surface modification or polymer grafting procedures to reduce and/or eliminate irreversible fouling.

The focus of this research is to characterize the influence of various electrospinning parameters on the formation of polySBMA fibers. PolySBMA has previously been studied as a solution to membrane fouling, but only as a grafted coating on preexisting membrane materials [7,25]. A more direct approach for creating membranes from this polymer is desired and electrospinning represents a promising approach. Electrospinning parameters including charge, solvent composition and polymer molecular weight were investigated to optimize the formation of nanofiber materials composed of polySBMA.

#### **MATERIALS AND METHODS**

#### **Materials**

N-(3-sulfopropyl)-N-methacryloxyethyl-N, N-dimethylammonium betaine (sulfobetaine methacrylate, SBMA) was purchased from Monomer-Polymer & Dajac Labs, Inc. (Trevose, PA). Sodium chloride, potassium chloride, and sodium azide, tris(hydroxymethyl) aminomethane were all purchased from Fisher Scientific (Pittsburg, PA).

Potassium persulfate, 99+%, was purchased from Acros Organics (Geel, Belgium). The dialysis membrane used was made of regenerated cellulose with a molecular weight cut-off of 12-14 kD and was purchased from Spectrum Labs, Inc. (Rancho Dominguez, CA). Ultrapure water (18.2M $\Omega$ ·cm) was obtained with a Millipore Synergy purification system (Billerica, MA) and used throughout.

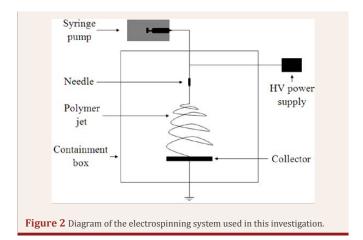
# Polymerization of SBMA

The SBMA polymerization procedures were adapted from the methods detailed by Zhang et al. [22]. Briefly, 0.05 moles of SBMA were dissolved in 100 mL pure water containing 5 mM potassium persulfate as an initiator and varying concentrations of KCl. The reaction was allowed to proceed for 5 hours under nitrogen protection. The polymerization reaction utilized different concentrations of KCl to control the final molecular weights. Dialysis was then performed for greater than 24 hours in ultrapure water to collect the polySBMA precipitate. For the polymerization completed in pure water, the polymer was precipitated out using ethanol. The molecular weights of the polymers were determined by size exclusion chromatography using a Waters 2690 Alliance high performance liquid chromatography (HPLC) system (Milford, MA) with a refractive index detector. The mobile phase buffer used for HPLC analysis was 0.05 M tris(hydroxymethyl) aminomethane in 1 M NaCl aqueous solution with 8 mM sodium azide. Each polySBMA was diluted to a 20 wt% solution before HPLC analysis. The molecular weights were calibrated using poly (ethylene oxide) standards run under identical conditions (Varian, Inc., Foster City, CA). For each molecular weight measurement, three independently polymerized polymer samples were characterized and each sample was run three times (n=9).

#### **Electrospinning**

The electrospinning apparatus consists of a high voltage power supply, a syringe pump, a metallic needle and a grounded collector. The system is fully enclosed in a plexiglass box for safety considerations. The different molecular weight polySBMA samples were dissolved in aqueous NaCl for electrospinning at a 1:1 volume ratio. To determine the influence of molecular weight on the resulting fiber characteristics, the amount of NaCl was fixed at 0.25 M. Then varying amounts of NaCl were used with one molecular weight polySBMA, in order to determine the effect of salt concentration on the size and morphology of the resulting nanofibers. The syringe pump had a constant flow rate of 0.5 mL/hr in all experiments. The optimal electrospinning voltage applied to the needle was determined to be 15 kV (data not shown) and then it was fixed for all experiments. The distance between the needle tip and collector was also fixed at 18 cm. The fibers were collected on glass microscope slides grounded by copper supports. A schematic of the electrospinning apparatus is shown in (Figure 2).





#### Scanning electron microscopy

The electrospun fibers were imaged using an FEI Quanta 600 Extended Vacuum Scanning Electron Microscope (SEM; Hillsboro, OR). Each sample was sputter coated with platinum for two minutes then photographed under high vacuum. A random sample of fiber diameters from each image were measured using Image J software [34]. For each electrospinning condition investigated, three independent nanofiber samples were imaged and the fiber diameter of twenty fibers was measured from each image (n=60).

#### Data analysis

Data are presented as the mean of all measurements from three independent samples plus/minus one standard deviation. Samples were identified as statistically significant from each other at a 95% confidence interval (p < 0.05). The statistical analysis calculations were completed with a one-way analysis of variance using Origin 8.5 software (OriginLab Corporation; Northampton, MA).

#### **RESULTS AND DISCUSSION**

# Molecular Weight of PolySBMA

To obtain polymers of different molecular weights, SBMA was polymerized with varying amounts of KCl in the reaction solution. According to Wang et al, the presence of KCl speeds up the polymerization rate by increasing the hydrodynamic radius thereby reducing the steric hindrance associated with accessing the polymerization site [35]. This results in a reduction of the molecular weights at increasing concentrations of KCl. The polymerization reaction was carried out using 0 M, 0.5 M, 1 M and 2M KCl concentrations. As the concentration of KCl was increased from 0 M to 2 M with all other reactions conditions fixed, the molecular weight of the polySBMA decreased from 474 kDa to 393 kDa as detailed in (Table 1). The polydispersity index for each reaction condition is also provided in (Table 1). While both values were higher than those found under similar reaction conditions [22], this was not a critical parameter for the focus of this investigation.

# Effect of Molecular Weight on Fiber Size

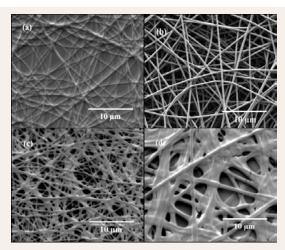
In order to determine the effect of molecular weight on fiber

size, electrospinning solutions were prepared for each polySBMA molecular weight as a 1:1 v/v ratio in aqueous 0.25 M NaCl. These solutions were then electrospun under the fixed conditions described in the Methods section. Representative SEM images of the electrospun fibers obtained for each molecular weight condition are shown in (Figure 3). In this figure it can be seen that as the molecular weight was increased, it resulted in larger diameter fibers. The two smaller molecular weights also appear to produce cleaner fibers with more uniform diameters. Once the molecular weight increased above  $\sim\!420$  kDa, the fibers exhibited a tendency to cluster and "melt" together at the intersection points between individual fibers. These observations can be attributed to the interrelated effects of an increased viscosity of the electrospinning solution which is caused by the simultaneous increase in the polymer molecular weight.

These parameters are well known to influence the resulting fiber characteristics [36]. The results suggest that under the electrospinning conditions investigated here, the molecular weights of  $393 \, \text{kDa}$  and  $420 \, \text{kDa}$  would be the most suitable for use as an electrospun filtration membrane. A random sample of fibers was measured from multiple SEM images to specifically quantify the differences in the final fiber diameter. As the molecular weight was increased from  $393 \, \text{kDa}$  to  $474 \, \text{kDa}$ , the average fiber diameter increased from  $328 \, \text{nm}$  to  $752 \, \text{nm}$ , as shown in (Figure 4). Furthermore, the mean diameter for each molecular weight was determined to be statistically significant from the next sample based on a 95% confidence interval (p<0.05). Based on the uniformity of the fiber diameter, polySBMA with a molecular weight of  $393 \, \text{kDa}$  was selected to investigate the influence of

**Table 1:** Molecular weight and polydispersity values for polySBMA polymerized under varying concentrations of KCl.

Conc. of SBMA (M)	Conc. of KCl (M)	Mn (kDa)	Polydispersity
0.5	0	474 ± 213	1.94 ± 0.33
0.5	0.5	422 ± 121	1.83 ± 0.20
0.5	1	420 ± 86	1.81 ± 0.08
0.5	2	393 ± 281	2.08 ± 0.14



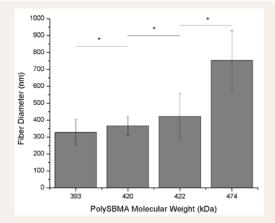
**Figure 3** Representative SEM images (~10,000x) of electrospun nanofibers formed from different molecular weight polySBMA samples: (a) 393 kDa, (b) 420 kDa, (c) 422 kDa, (d) 474 kDa. All other electrospinning parameters including NaCl concentration (0.25 M) were fixed.

the salt concentration in the electrospinning solution. It should be pointed out that it is possible to improve the fiber uniformity and quality for the higher molecular weight polySBMA samples by varying additional experimental parameters including the concentration of the polymer (and solution viscosity) and the applied current.

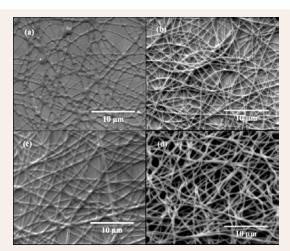
## **Effect of Salt Concentration on Fiber Size**

It has previously been shown that the concentration of salt in the electrospinning solution can impact the fiber characteristics because it influences the electrical conductivity of the solution [37]. Therefore, the amount of salt in the electrospinning solution was varied to determine its influence on the resulting polySBMA fibers in this system. NaCl concentrations of 1 M, 0.5 M, 0.25 M, and 0.17 M were investigated with the smallest molecular weight SBMA polymer (393 kDa). Representative SEM images of the fibers formed using the different NaCl concentrations are shown in (Figure 5). In this Figure, it can be seen that the concentration of NaCl in the electrospinning solution clearly affected the nanofiber characteristics. At the largest salt concentration the fibers are less defined and shown some beading effects, especially at fiber intersection points. The other salt concentrations appear to produce more uniform fibers. The fiber diameter also appears to be larger at the lowest salt concentrations as compared to the higher concentrations.

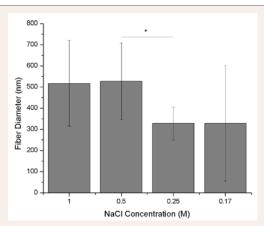
The average fiber diameter was measured for a representative group of fibers to quantify the effect of the NaCl concentration. These quantitative results are shown in Figure 6. The fiber diameters were nearly identical between the 1 M and 0.5 M concentrations and between the 0.25M and 0.17 M concentrations. However, the fiber diameter differences between the larger two and the smaller two salt concentrations were statistically significant. These results suggest that any of the three lowest salt concentrations produce fibers that may have use as a standalone filtration membrane. As before, it is possible that the 1 M NaCl solution can also be used to form uniform fibers if additional electrospinning parameters are modified.



**Figure 4** Mean ± standard deviation of the measured fiber diameters for nanofibers formed from different molecular weight polySBMA samples. All other electrospinning conditions including NaCl concentration (0.25 M) were fixed. At least 20 measurements were taken for each of 3 independent samples (n=60). \*Represents a statistically significant difference between the samples being compared (p<0.05).



**Figure 5** Representative SEM images (~10,000x) of 393 kDa poly SBMA electrospun nanofibers from solutions with varying concentrations of NaCl: (a) 1 M NaCl, (b) 0.5 M NaCl, (c) 0.25 M NaCl, (d) 0.17 M NaCl. All other electrospinning parameters were fixed.



**Figure 6** Mean ± standard deviation of the measured fiber diameters for 393 kDa poly SBMA electrospun in solutions with varying concentrations of NaCl. All other electrospinning conditions were fixed. At least 20 measurements were taken for each of 3 independent samples (n=60). \*Represents a statistically significant difference between the samples being compared (p<0.05).

#### **CONCLUSION**

In this work, optimal conditions for electrospinning polySBMA nanofibers were determined. While keeping all other electrospinning parameters constant, it was shown that as the molecular weight of the polySBMA was increased, so was the fiber diameter. However, the maximum molecular weight that resulted in the formation of uniform, smooth nanofibers was ~420 kDa indicating that this is a critical cut-off value. The effect of salt concentration in the electrospinning solution was also demonstrated. While each individual salt concentration did not result in a statistically significant difference in the measured fiber diameter like the molecular weight did, a critical salt concentration was also identified. The fibers began to break down when the salt concentration was increased above 0.5 M. The results of this study identified two critical cut-off parameters that must be met in order to electrospin uniform polySBMA nanofibers for filtration applications. Current efforts are focused



on characterizing the mechanical and nonfouling properties of these materials as a function of the electrospinning parameters

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