FMRI RET 2015-Characterization of ATO and Cobalt Doped ATO Polymeric Solutions for Nanofiber Fabrication

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Abstract

Electrospinning of polymers allows for thin, porous and light nanofiber layers to be applied in various ways. The ultimate goal of this project is to electrospin a nanofiber layer of polymer with antimony doped tin oxide (ATO) and cobalt oxide (CoO) to be used as an intermediate or top coating layer of a photovoltaic (PV) cell. Various structures dip coated in ATO and cobalt doped ATO have shown improved efficiency and thermal stability. Preliminary research this summer has focused on analyzing UV/VIS absorbance of various concentrations of polystyerene (PS), d-limonene, ATO and CoO solutions using the spectrophotometer. These initial investigations have focused on collecting data in the visible range to determine if there is any shift in the spectrum due to cobalt doping.

Background

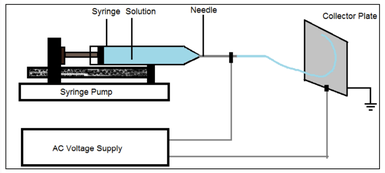

Figure 1: Illustration of the electrospinning equipment [7]

Electrospinning of polymeric solutions can produce nanofiber layers by exposing the polymeric solution to an electric field (Figure 1). The electrostatic force eventually breaks the surface tension of the solution and a jet of extremely thin solution is released. This jet will land on a ground plate (metal collector). If solution parameters are set correctly, the solvent will evaporate leaving thin fibers.[1]

Electrospinning dates back to a 1934 patent by the company Formalas. Within the last two decades the field of electrospun nanofibers has expanded signficantly.[3]

Electrospun nanofibers are being studied for uses in life sciences (tissue engineering, drug delivery, wound dressings), filter media, micro/nano devices, LCDs, sustainable sources, nano sensors (thermal, piezo, biochemical), military and cosmetic applications.[1,2,3] The ultimate goal of this project is to electrospin a nanofiber layer of polymer with antimony doped tin oxide (ATO) and cobalt oxide (CoO) to be used as an intermediate or top coating layer of a photovoltaic (PV) cell. Current research in solar cells is attempting to make them more efficient and find potentially less expensive production materials.[4] ATO is antimony (Sb) doped tin oxide (SnO_2) in a toluene solution. SnO_2 is best controlled when doped in a solution, like ATO. The Sb has been found to increase the electrical conductivity of the SnO_2. Other properties include low resistivity, over 80% optical transparency, IR reflection and relatively low cost.[5] Adding CoO to ATO has been shown by the AMBIR lab to enhance the electrical conductivity and reflectivity of the coating.[6]

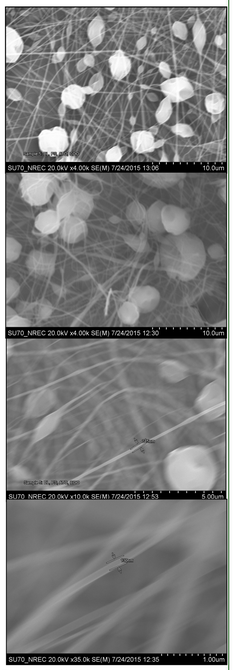

Figure 2: SEM pictures of nanofibers made of DL, PS, ATO and CoO.

Objectives

The ultimate goal of this project is to electrospin a nanofiber layer of polymer with antimony doped tin oxide (ATO) and cobalt oxide (CoO) (Figure 2) to be used as an intermediate or top coating layer of a PV cell. Preliminary research this summer has focused on analyzing UV/Vis absorbance of various concentrations of PS, d-limonene, ATO and CoO solutions using the spectrophotometer. These initial investigations have focused on collecting data in the visible range to determine if there is any shift in the spectrum due to cobalt doping.

Approach

Preparation of Solutions

Solutions were designed using a percent by weight ratio. The following is a table of solutions and their percent by weight combinations.

% by weight of each solution	DL	ATO	CoO	DL, PS	DL, ATO	DL, CoO	DL, ATO, CoO	DL, PS, ATO	DL, PS, CoO	DL, PS, ATO, CoO	DL, PS, ATO, CoO	ATO, CoO
d-limonene (DL)	100	0	0	81	84	84	32	75	71	76	71	0
Polystyrene (PS)	0	0	0	19	0	0	0	17	18	19	18	0
ATO	0	100	0	0	16	0	34	8	0	3	4	50
CoO	0	0	100	0	0	16	34	0	12	3	8	50

Table 1: Solutions tested and their compositions in percent by weight.

The d-limonene, ATO and CoO liquids were measured using micropipettes. Polystyrene was measured accurately to 0.01 of a gram. All components of the solutions were combined in a glass vial with a foil lined airtight cap and then stirred for an hour using a magnetic stir bar (6mm x 25mm) on a magnetic stirring hot plate (no heat was added) as in Figure 3. Additionally, before any kind of testing or electrospinning solutions were mixed again using a vortex for about 1 minute, or until the CoO is completely blended into the solution.

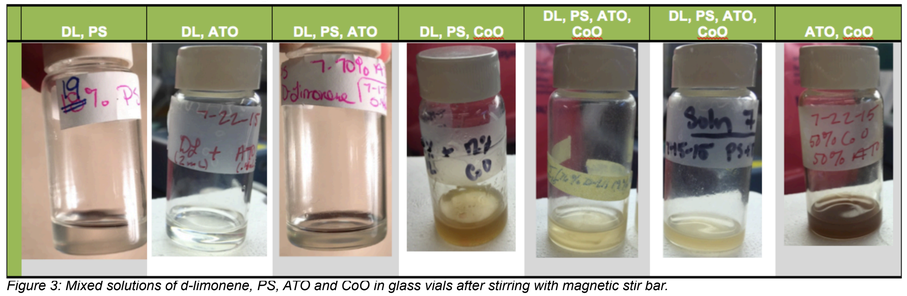

Figure 3: Mixed solutions of d-limonene, PS, ATO and CoO in glass vials after stirring with magnetic stir bar.

UV/Vis Spectrophotometer

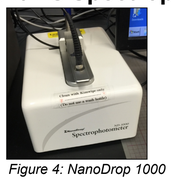

Figure 4: NanoDrop 1000

Measurements were recorded using the NanoDrop 1000 Spectrophotometer (Figure 4). The equipment was cleaned, initialized and blanked using distilled water. Each sample was mixed again using a vortex for about 1 minute, or until the CoO was completely blended into the solution. Then 2 μL of each sample were measured using a micropipette and placed on the stage. Between each sample tested water and pure d-limonene were used to clean the stage and ensure nothing was left behind. The spectrophotometer measures absorbance from 220 nm to 750 nm and displays it in a graph of absorbance versus wavelength.

Electrospinning Solutions into Fibers

The experimental set up, see Figure 1, requires a voltage regulator, a syringe pump, and a metal collector plate. A base of aluminum foil is wrapped around the metal plate so that the fiber sample can be easily removed without damage. The collector plate is set 20cm from the needle. The AC power supply is connected to the collector plate via an alligator clip. These parameters were determined in previous studies by the AMBIR lab as ideal parameters to produce nanofibers with less beading and dripping.[7]

To ensure a homogeneous solution, each sample is mixed in the vortex for one minute or until CoO is completely blended into the solution. Once the solution is properly mixed, the syringe is filled with about 1 mL of solution. Then a blunt needle is put on the syringe and all air is released from the syringe. The syringe is loaded into the syringe pump and an infusion rate of 20 μL/min is set.

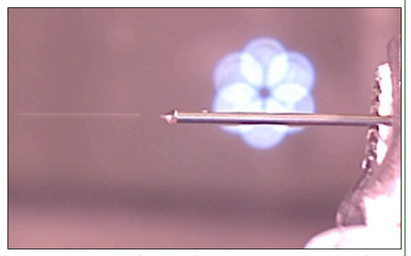

Figure 5:150x magnification of the Taylor cone at the tip of needle with visible jet being released.

The power supply is set to 20 kV. Once the power supply is turned on this forms an electric force field between the solution and the collector plate. When the solution in the syringe is pushed out, the solution becomes charged by the high voltage supply, a cone appears at the tip of the needle (Taylor cone), intermolecular forces within the solution begin to break and using a magnifying lens, very thin fibers can sometimes be seen extruding from the cone (Figure 5). The extracted fibers land on the collector plate.

Each sample was allowed to run for an hour to ensure a large enough sample to analyze via scanning electron microscope. A good indicator fibers have formed is a white layer on top of the aluminum foil, however, in order to verify the formation of nanofibers it is necessary to evaluate the sample using a scanning electron microscope (see Figure 2 for example images).

Conclusions

Solution	Peak absorbance wavelength (nm)
DL	265.75
CoO	267.5
DL, ATO	273.25
DL, CoO	274.75
DL, PS	277.75
DL, PS, ATO, CoO (3%)	280.75
DL, PS, ATO, CoO (8%)	283.75
DL, PS, ATO	287.25
DL, PS, CoO	291
ATO	298
ATO, CoO	301.5

Table 2: Wavelengths of peak absorbance

The spectrophotometer measures absorbance from 220 nm to 750 nm and displays it in a graph of absorbance versus time. The results of each sample were analyzed in two ways. The first was to analyze the wavelength of the peak absorbance of each solution (See Table 2). The second was to evaluate the complete graphs and to compare other wavelengths where absorption was observed or not observed (See Figures 6 and 7).

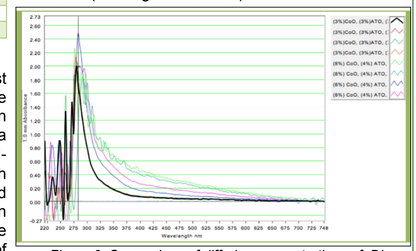

Figure 6: Comparison of differing concentrations of DL, PS, ATO, CoO solutions.

First, the wavelengths of the highest absorption for each solution were compared (see Table 2). A comparison of the samples shows that there is a difference in optical reactivity when d-limonene and polystyrene are mixed with ATO and CoO. Furthermore, it did appear there was a small difference in the wavelengths of peak absorbance based on percentage (3% vs. 8%) of ATO and CoO in the solution. Further studies should be done to check for a greater shift with increased CoO doping.

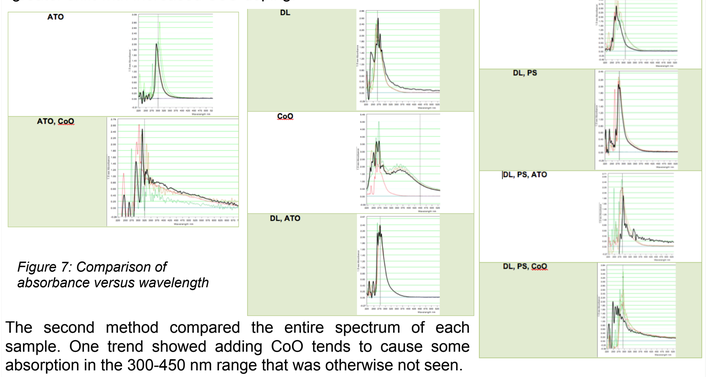

Figure 7: Comparison of absorbance versus wavelength

The second method compared the entire spectrum of each sample. One trend showed adding CoO tends to cause some absorption in the 300-450 nm range that was otherwise not seen.

This is also supported by Figure 6 which compares the DL, PS, ATO and CoO solutions where the concentrations of CoO differ and the absorbance increases in that range as CoO concentration increase. Further studies should be done exploring why CoO absorbs in the 300-450 nm range and its impact on PV cells. Additionally, studies should be done on the spun nanofibers to see if they too show these same patterns of absorption.

Acknowledgements

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Referenced Resources

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