Reactive Electrospinning of Xerogel Composites

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Objective:

To investigate a new technique for ceramic metal oxide membrane production via reactive electrospinning

Sol-gel Process

In the traditional sol-gel process, a metal alkoxide (i.e. $Mg(OCH_3)_2$) solution is partially hydrolyzed to form a reactive monomer followed by the condensation of the monomers to form colloid structures (sol formation). Further hydrolysis leads to polymerization of the colloids (gel formation). Produced gels then undergo various drying techniques to produce desired products, including ceramic fibers.

$$Mg(OCH_3)_2 + H_2O \rightarrow Mg(OH)(OCH_3) + CH_3OH$$

Partial Hydrolysis

$$Mg(OH)(OCH_3) + H_2O \rightarrow Mg(OH)_2 + CH_3OH$$

Condensation

$$Mg\text{-}OCH_3 + HO\text{-}Mg\text{-} \rightarrow \text{-}Mg\text{-}O\text{-}Mg\text{-} + CH_3OH$$

Polymerization

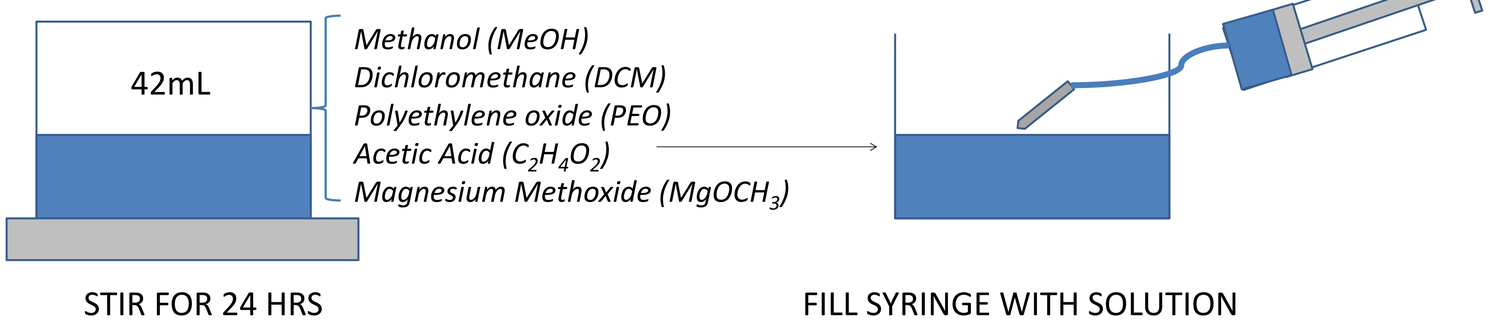
$$\text{-}Mg\text{-}OH + HO\text{-}Mg\text{-} \rightarrow \text{-}Mg\text{-}O\text{-}Mg\text{-} + H_2O$$

Reactive Electrospinning

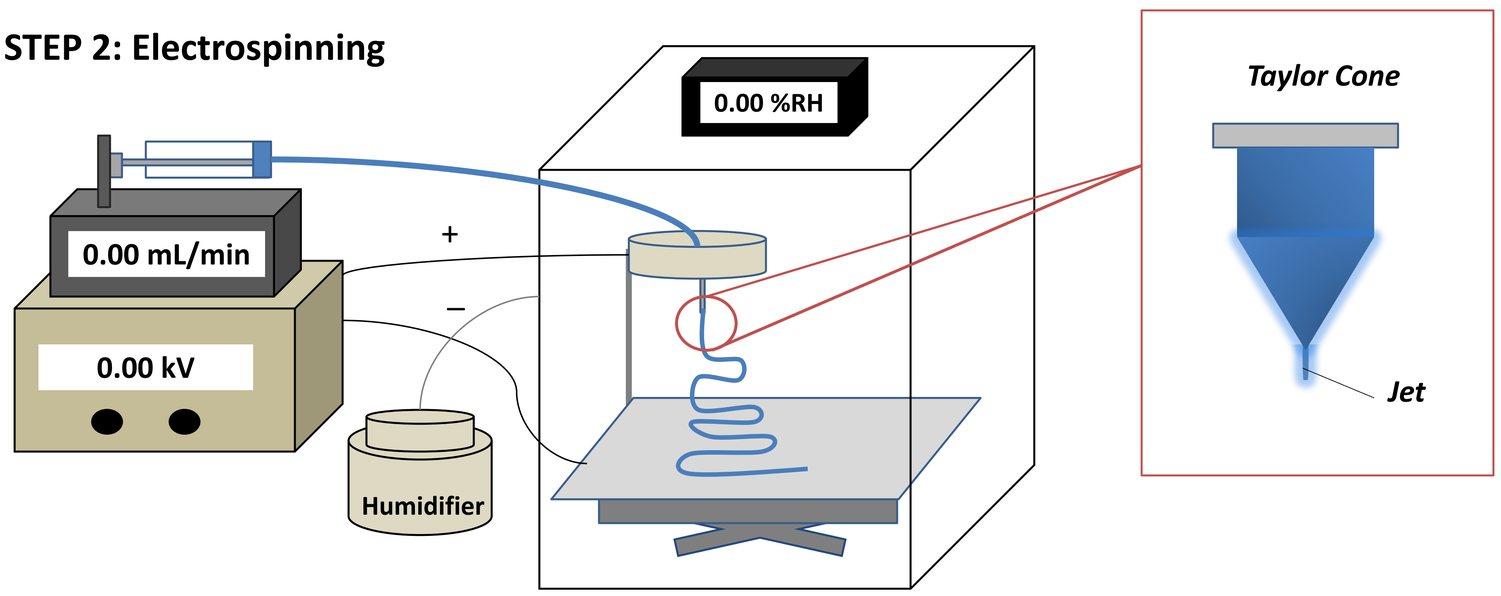
During reactive electrospinning, an unreacted metal alkoxide is electrospun in a humid-controlled chamber. The moisture in the ambient initiates the identical sol-gel process outlined above, directly producing ceramic fibers. Utilizing the mass transport (i.e. solvent-solvent exchange) taking place in the jet formed during electrospinning, the reaction occurs quickly.

Experimental Procedure

STEP 1: Metal Alkoxide Precursor



STEP 2: Electrospinning

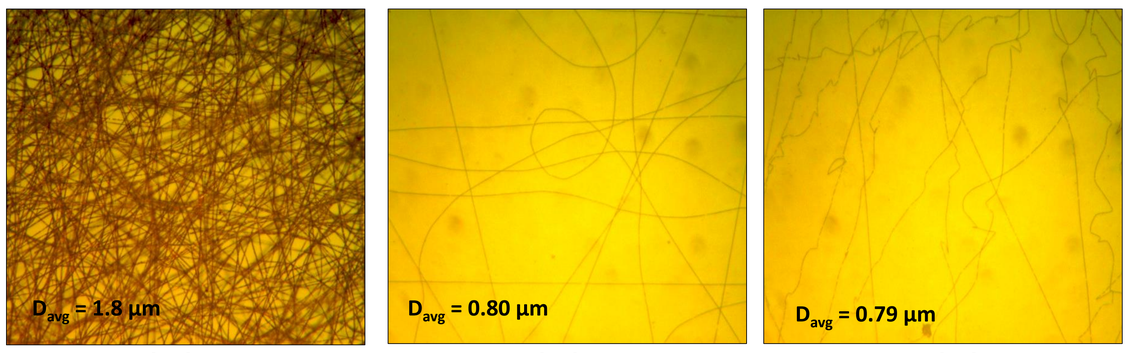


Variables

Substrate (PEO) weight percent, solvent ratio, flow rate, voltage supplied and chamber humidity

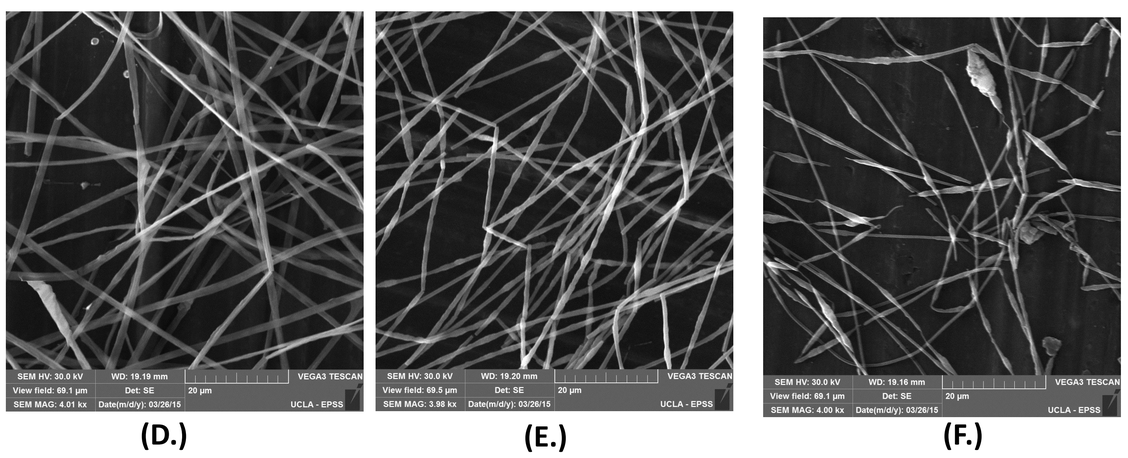
Results

Solution: 80:20 MeOH:DCM, 0.25wt% PEO, 0.1wt% $C_2H_4O_2$, 12mL $MgOCH_3$ electrospun at flow rates of 0.02-0.3 mL/min and supplied voltage of 7-8 kV



(A.) **(B.)** **(C.)**

Micro slide images (above) of solution electrospun at (A.) 0%, (B.) 30%, (C.) 50% relative humidity.



(D.) **(E.)** **(F.)**

Scanning Electron Microscope images (above) of solution electrospun at (D.) 0%, (E.) 30%, (F.) 50% relative humidity.

Conclusion

For this project, various solutions varying in PEO weight percent (0.2-0.5 wt%) and solvent ratio were tested at varying supplied voltages (1-14 kV) and flow rates (0.01-1.0 mL/min). Doing so provided as preliminary data needed to understand variables affecting reactive electrospinning. The above solution demonstrated the best results based upon jetting performance. ToupView® software was used to measure fiber diameter. A clear relationship between humidity and fiber diameter is yet to be determined.

Future Work

In analyzing the obtained SEM images, it was noted that water is present in the fibers produced. To avoid contact with water prior to entering the humidity-controlled chamber, the solution will be prepared inside a glove box purged of ambient air and filled with dry air. Additionally, infrared spectroscopy will be used to analyze the chemical composition of the produced fibers to verify the presence of the Mg-O complex desired.