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Investigation of the electrospinnability of polymer solutions using tubeless siphoning

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Electrospinning

1. Electrospinning of polymer solution

- Electrospinning is a unique technique for fabrication of **nanofibers**
- Constant **elongational flow** of polymer solution is maintained from a reservoir
- An **extensional force** is applied by high electrical voltage
- A conductive substrate, normally grounded, serves as a **collector**

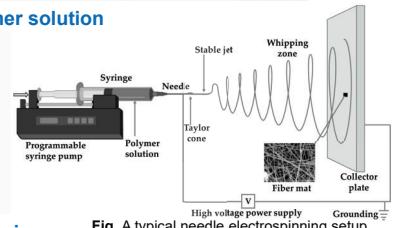
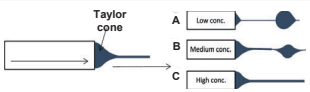
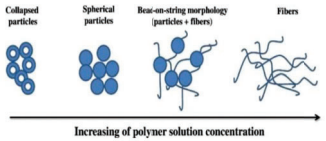


Fig. A typical needle electrospinning setup

2. Challenges of electrospinning

- Electrospinnable range specific to polymer
- Specific to molecular weight
- Polymer solution concentration
- Costly way of determining electrospinnable range





Tubeless siphoning

1. Tubeless siphoning of polymer solution

- Tubeless siphoning, a unique property of non-Newtonian fluid, is a pure **extensional flow**
- Unlike Newtonian fluid, siphoning action continues even when the siphon nozzle is raised above the free surface of the fluid
- Viscoelastic stresses, resulting from stretching of the polymer molecules in solution, support the weight of the column

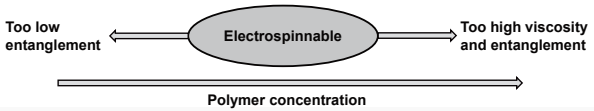
Fig. Tubeless siphoning of polyethylene oxide (PEO) solution

2. Factors influence Tubeless siphoning

- Molecular weight and concentration of polymer solution
- Entanglement of the polymer chains in the solution
- Applied extensional force

Motivation and objectives

- Electrospinning is possible for a concentration range of a specific polymer
  - Two low concentration ⟶ lack of chain entanglement ⟶ Does not form continuous fiber
  - Two high concentration ⟶ too high chain entanglement to draw the solution



- Tubeless siphoning for a polymer solution also possible for a definite range of concentration
  - Two low concentration ⟶ lack of chain entanglement ⟶ Does not form siphon column
  - Two high concentration ⟶ too high chain entanglement to form a siphon column
- Both the electrospinning process and tubeless siphoning involve high extensional flow

Objective of this work

- Establishing a correlation between siphonable range and electrospinnable range for a polymer solution with different molecular weight
- Predicting electrospinnability of a polymer solution from tubeless siphoning
- Reducing the costly process of determining electrospinnability

Materials and methods

1. Tubeless siphoning of PEO solutions

- A series of concentrations (0.05 -10 w/w %) of PEO solutions with MW 400k, 900k, and 5000k were investigated
- Backlight imaging technique was used for measuring the siphon height for each solutions

2. Electrospinning of PEO solutions

- A needle electrospinning setup was used for electrospinning of all PEO solutions
- A constant flow rate, high electric voltage, and needle tip-to-collector distance were maintained

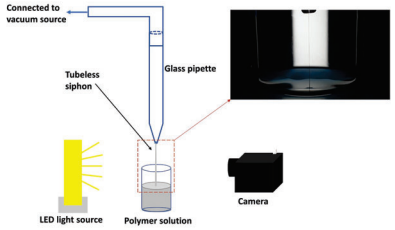


Fig. Tubeless siphoning setup for PEO solution with the arrangement of backlight imaging technique

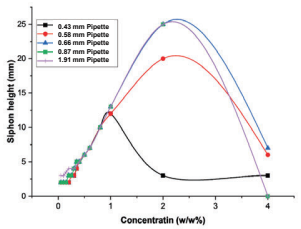
Process parameters of electrospinning		
Flow rate (ml/h)	Voltage (kV)	Needle tip-to-collector distance (cm)
0.5	15	14

Results and discussion

1. Effect of concentration of PEO solution (400k) on siphon height

- Maximum siphon height was observed for 2% (w/w) solution
- Siphonable range : 0.25-6% (w/w)
- Pipette with diameter at or above 0.66 mm shows maximum siphon height
- Consistency of data for multiple runs (3-5) is remarkable

Force at pipette tip		
Pipette diameter (mm)	Cross sectional area in m² (10^{-7})	Force in N (considering pressure -0.60 bar)
0.43	1.457	0.0087
0.58	2.628	0.0158
0.66	3.413	0.0205
0.87	5.959	0.0358
1.91	28.673	0.1720



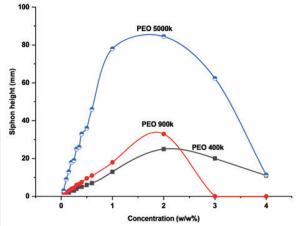


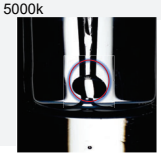
Fig. Suction pressure: ~ -0.60 bar, pipette diameter: 0.87 mm

2. Effect of molecular weight

- Siphon height increases significantly with increasing MW of PEO
- Siphon height for PEO 5000k is four times higher than that of PEO 400k
- Different maximum siphon heights concentration for different MW of PEO (400k: 2%, 900k: 2%, 5000k: 1.5%
- Siphon heights for all runs (3-5) are remarkably consistent

3. Siphonable range of PEO solutions

- Jelly fish formation for PEO solutions with concentration at or above 2 % for PEO 400k and 900k, and 1.5 % for PEO 5000k
- Siphon height decreases significantly at jelly fish formation region
- No siphoning at concentration 6 % for PEO 400k and 900k, and 5 % for PEO 5000k



Jell fish formation

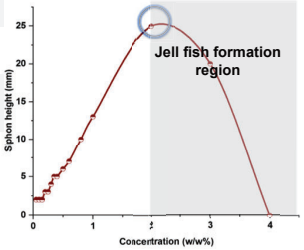


Fig. PEO 400k, suction pressure: ~ -0.60 bar, pipette diameter: 1.91 mm

Results and discussion

4. Electrospinnability of PEO solutions

A. Electrospinnability of PEO solutions (MW: 400k)

2% solution: Beaded fibers
4% solution: Beaded fibers
6% solution: Uniform fibers

B. Electrospinnability of PEO solutions (MW: 900k)

2% solution: Beaded fibers
3% solution: Beaded fibers
5% solution: almost uniform fibers (fewer beads)

C. Electrospinnability of PEO solutions (MW:5000k)

2% solution: Beaded fibers
3% solution: Beaded fibers
5% solution: Uniform fibers

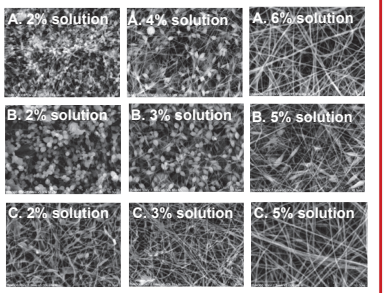
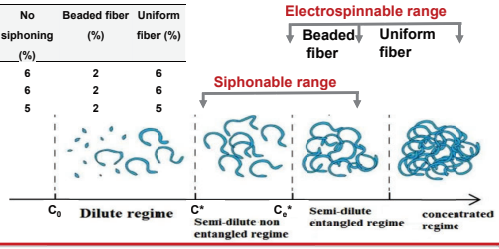


Fig. SEM images of electrospun fibers of PEO solution A: PEO 400k, B: PEO 900k, C: PEO 5000k

5. Correlation between siphonable range and electrospinnable range

Polymer	Jelly fish formation (%)	No siphoning (%)	Beaded fiber (%)	Uniform fiber (%)
PEO 400k	2	6	2	6
PEO 900k	2	6	2	6
PEO 5000k	1.5	5	2	5



Conclusions

- PEO solution exhibits tubeless siphoning within the range of 0.25 and 6%
- 'Jelly fish' structure along siphon column at 2% for PEO 400k and 900k, and 1.5% for PEO 5000k
- No siphoning was possible at 6% for PEO 400k and 900k, and 5% for PEO 5000k
- For PEO 400k and 900k: beaded fiber at concentration 2-5%
  bead free fibers at or above 6%
- For PEO 5000k: beaded fiber at concentration 1.5-4%
  bead free fibers at 5%

Future work

- Correlation between siphonable range and electrospinnable range for other water-soluble polymers such as polyacrylamide (PAM) and PAM-co-DADMAC
- Effect of addition of salts, surfactants, and PEG, temperature, pressure and solvents on tubeless siphoning
- Measuring extensional viscosity of polymer solutions from maximum siphon height measurement
- Using backlight imaging technique to correlate siphon pattern with solution concentration and pipette diameter

References

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