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Responsive Polymer Grafts for Patternable Self-Oscillating Gels

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MOTIVATION

- Polymers that experience drastic changes in their physical properties in the presence of environmental stimuli (temperature, pH, light, humidity, etc.) are called responsive polymers.
- Responsive materials hold great potential as sensors and actuators that function autonomously. For example, pumps or stirrers for microfluidic devices in lab-on-a-chip applications.

BACKGROUND

- Self-oscillating gels are a class of responsive polymer materials that exhibit autonomous swell and deswell oscillations. These gels are driven by the Belousov-Zhabontinsky (BZ) reaction and operate by converting chemical energy to mechanical behavior through patterning of BZ gel patches.
- Recently a printable BZ gel based on gelatin was developed. Gelatin, however possesses some challenges for long term implementation:
  - It degrades in BZ solution over time (12-24hrs).
  - Below 32 °C, it becomes miscible.
  - Above 32 °C, it becomes immiscible.
  - This thermoresponse property could potentially be harnessed to produce a thermally reversible gel that could be patterned through printing.
  - There are many types of copolymers: random, graft, comb, and di-block.
  - PNIPAm graft type copolymers should be able to be designed to produce reversible physical crosslinks (leading to gelation).

PRIMARY AIM

Develop a synthetic, thermally reversible gel using responsive graft copolymers, that can be easily patterned through printing technologies.

EXPERIMENTAL / PROCEDURE

- A procedure from the literature for the synthesis of PNIPAm grafts was adopted.
- N-isopropylacrylamide (NIPAm) was polymerized with amine functionality. The amine was then converted into a vinyl-capped PNIPAm using an amide condensation reaction between the amino groups in PNIPAm and N-acryloxysuccinimide (NAS).

Procedure Summary

- NIPAm was mixed with 2-aminooethanethiol (AESH) in N,N-Dimethylformamide (DMF).
- The solution was freeze-pump-thawed to remove any oxygen that might prevent the polymerization.
- The oxygen-free solution was water-bathed at 75 °C with stirring.
- After heating for 15 hours, the product was precipitated in acetone/hexane solution.
- The precipitated powders were mixed with NAS in DMF solution.
- After keeping the solution at 4 °C for 2 days, the product was precipitated in acetone/hexane solution.
- The precipitation procedure in diethyl ether as specified, produced unusable product.
- Instead, the product, either amine capped or vinyl capped PNIPAm, was dissolved in sufficient amount of acetone and added drop wise into hexane while vigorously stirring.
- The optimal ratio of PNIPAm to hexane solution was PNIPAm/hexane=1:100.
- Lower ratio resulted in sticky precipitation.
- The formation of amine-capped PNIPAm and vinyl-capped PNIPAm were verified using IR Spectrum.
- The average molecular weight of the grafted materials was determined by the end group tiration with acetic acid/perchloric acid.

CONCLUSIONS / FUTURE WORK

- Vinyl-capped PNIPAm grafts with different length was successfully synthesized.
- More characterization is required to confirm the functional group changes.
- Next steps are to polymerize these grafts with a backbone polymer to produce a thermally reversible gel and to incorporate ruthenium complex to run the BZ reaction for its autonomous characteristic.

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