Synthesis of Liquid Crystal Elastomer Threads for the Creation of Self-Tying Knots

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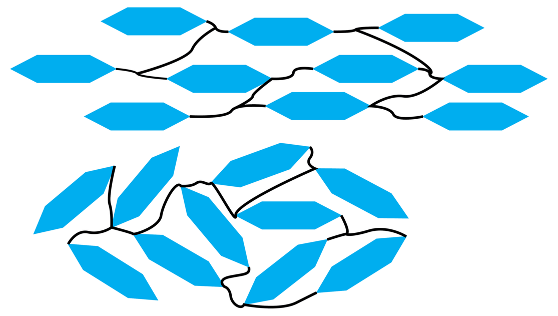
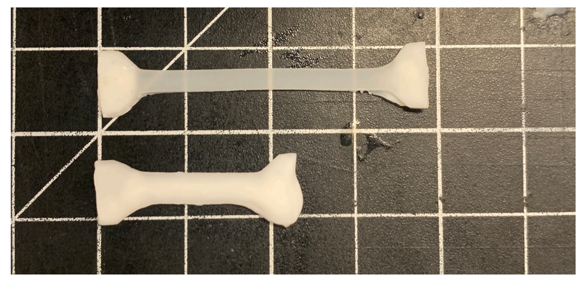
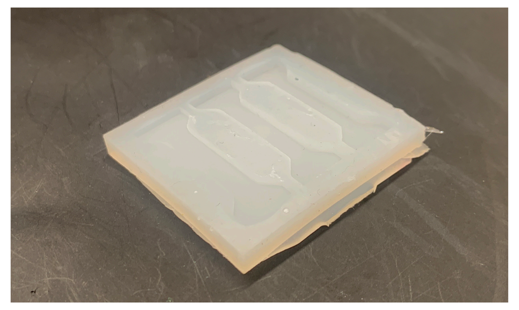
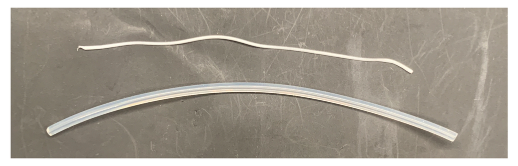
Background

- Liquid Crystal Elastomers (LCEs) are polymers made of slightly crosslinked liquid crystal networks
- LCEs can retain shape memory and actuate when subjected to an external stimulus, usually heat or light.
- LCEs have actuation properties because of their anisotropy
  - Nematic (aligned/stretched) state vs. isotropic (unaligned/condensed) state
- This research aims to create LCE strands that exhibit effortless actuation and self-tightening upon exposure to light.

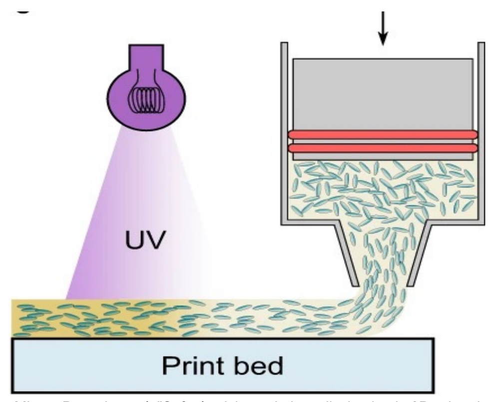
Methods (LCE Synthesis)

Chemical (in grams)	1:1	1:1.4	1:1.8	1:2.2
RM82	2	2	2	2
Toluene	0.62	0.62	0.62	0.62
EDDET	0.111	0.1555	0.1999	0.2443
PETMP	0.5952	0.8333	1.0714	1.3095
HHMP	0.00472	0.00472	0.00472	0.00472
DPA	0.00648	0.00648	0.00648	0.00648

One Pot Method for LCE Synthesis
RM82 monomer dissolved in toluene at 90° and then mixed with EDDET, PETMP, and HHMP. The solution was cooled, and additives were incorporated. DPA in a 1% w/w toluene solution was added, and the mixture was poured into a mold. Strands were formed by injecting the solution into a tube. After overnight curing, the polymer was removed, and residual toluene evaporated at 90°C. Chemical quantities were adjusted based on the desired dicell to thiol ratio.

RM257:EDDET Molar Ratio	EDDET Mass (g)
1.02:1	3.036
1.10:1	2.816
1.15:1	2.693
1.20:1	2.581
1.25:1	2.478
1.50:1	2.060

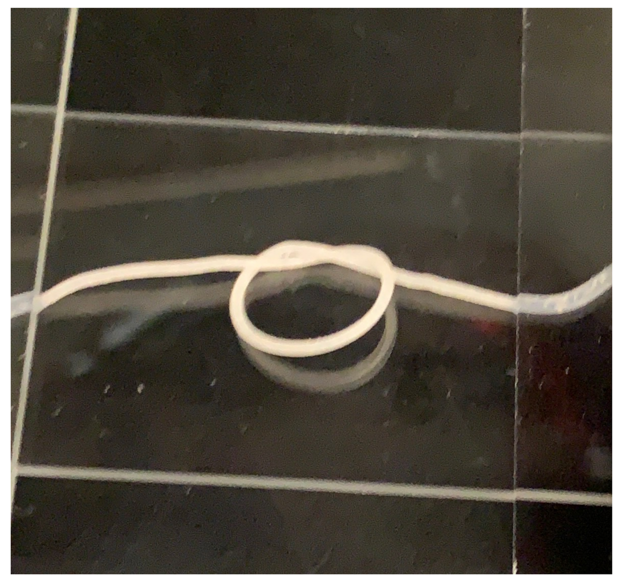


Mistry, Devesh, et al. "Soft elasticity optimises dissipation in 3D-printed liquid crystal elastomers." *Nature Communications* 12.1 (2021): 6677.

Oligomer Method
10g RM257 monomer dissolved in 125mL Methylene Chloride in a flask. EDDET mixed separately with 4 drops of DBU, amount adjusted based on desired RM257:EDDET ratio. Gradually added to flask, stirred for 16 hours. Added 0.1g BHT, and heated at 90°C for 12 hours, yielding an oligomer. Approximately 2g oligomer added to a vial at 90°C. 4% w/w methylene chloride and 2% w/w DMPA mixed in. Additives included. Oligomer loaded into a syringe, attached to an 18-gauge nozzle covered with foil. Syringe pump slowly extruded the oligomer, heated nozzle with a heat gun for easier extrusion. Extruded strand immediately cured with UV light. Strands separated and cured under UV light.

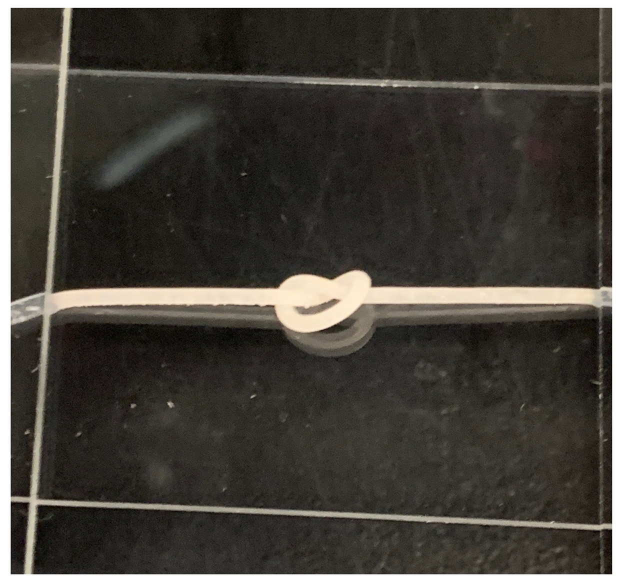
Results

LCE Strand IR-Light Tightening Proof of Concept



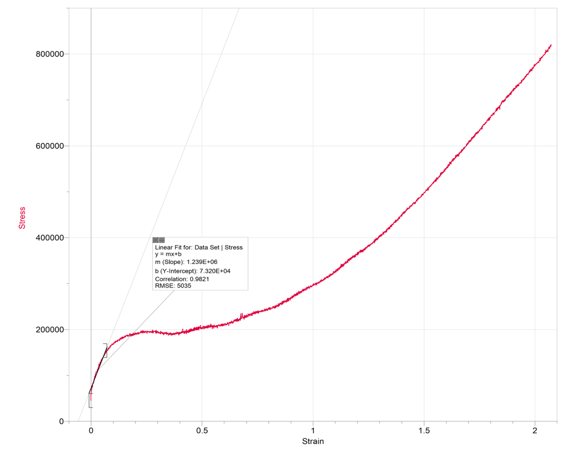
Pre IR-Light

A strand of LCE was loosely tied into an overhand knot shape. It was then clamped on both ends and heated using an IR light to test if the knot could tighten using only light as an outside stimuli.

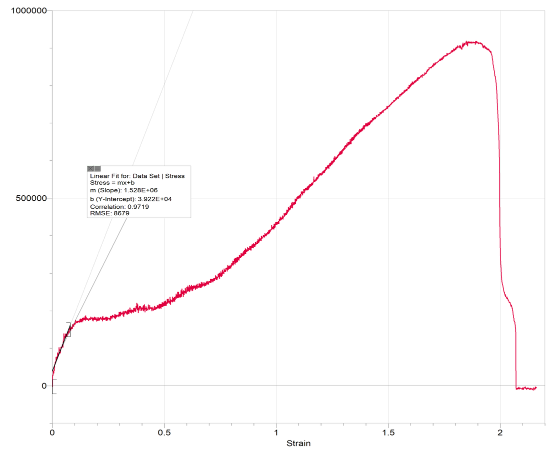


Post IR-Light

Young's Modulus Stress vs. Strain curve



Plain Sample
Slope: 1.239E+06

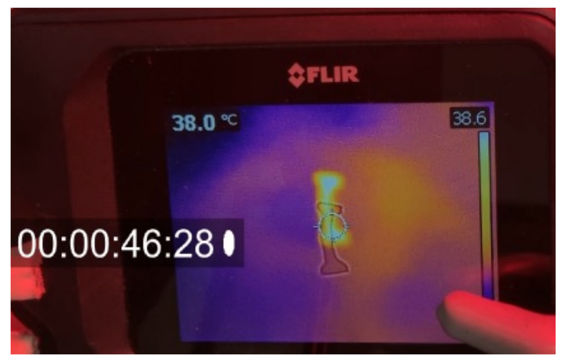


CNT Sample
Slope: 1.582E+06

A dogbone sample was placed in an INSTRON Universal Testing Machine, which measures the force on a load cell. The sample was stretched to the breaking point to measure the force needed to stretch the sample. The data was plotted in a stress vs. strain graph.

Working Temperature (Approximate TNI)

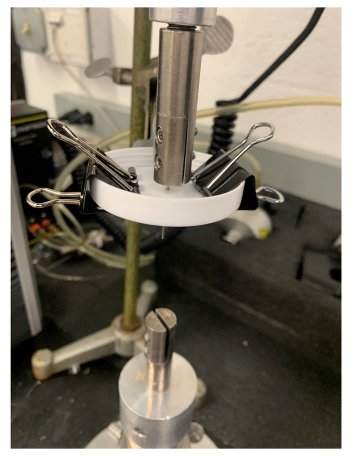
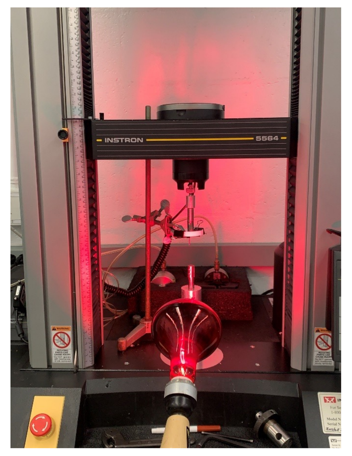
Plain Sample



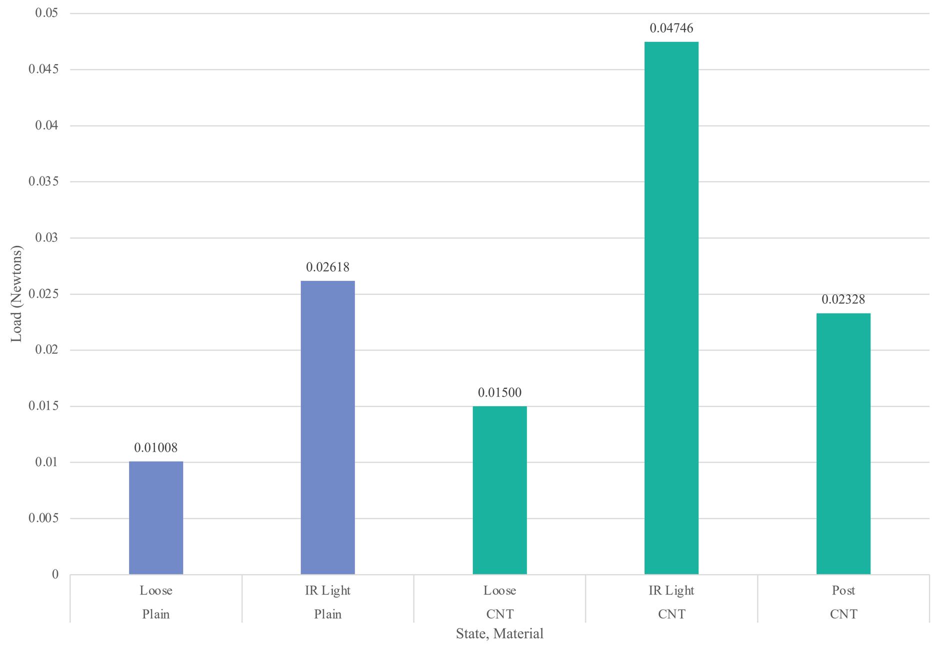
CNT Sample

A dogbone sample was stretched approximately 80% of its original length. The sample was placed on a white background. The sample was heated using an infrared light until full actuation was added. An infrared/temperature camera was used to record the temperature of the sample and the time needed to achieve actuation.

Knot Friction Force Test


A syringe needle was placed into the upper clamp of an INSTRON Universal Testing Machine. Using a lab beaker clamp and stand, a piece of plastic with a small hole the same diameter of the needle was placed right below the needle. The INSTRON was used to insert the needle into the hole. A stand of LCE was loosely knotted around the needle and the ends were clamped. The entire setup, depicted below, was used to measure the force required to retract the needle. The LCE knot was actuated using an IR light over a 2 minute period, tightening around the needle. The needle was retracted once more while the light remained on, with the force needed for retraction recorded. The two forces recorded were compared.



Conclusion

One-Pot Experimentation

- Most reactive One-Pot samples have a 1:1.18 dicell to thiol group ratio
- Decreasing the ratio increases crosslinking, resulting in less elastic LCE with enhanced actuation strength
- Reduced ratio leads to a significant increase in TNI temperature, suggesting higher energy requirement for actuation

Oligomer Experimentation

- Altering RM257 to EDDET ratio in oligomer method shows increased EDDET content leads to greater crosslinking, resulting in more reactive but less elastic LCE strand
- Higher EDDET content in formula makes extrusion of the strand more challenging, balancing increased actuation strength with difficulties in extrusion

- **One-Pot approach is more manageable compared to the oligomer method, chosen for subsequent tests.**

Young's Modulus & TNI Testing

- Addition of GO and CNCs did not significantly alter samples, while CNTs effectively reduced TNI and enhanced actuation efficiency
  - CNTs' black color and shape enhanced heat absorption from IR light
  - CNT samples are stiffer, less elastic, and exhibit higher actuation force in knot friction test

LCE Strand IR-Light Tightening Proof of Concept

- The proof of concept indicates that LCE strands have the ability of forming self-tightening knots because of their shape memory

Future Applications

- Potential applications of self-tying knots in knot-based actuators demonstrated in proof of concept
- Infrared (IR) light induction of movement is promising for practical applications.

Future Research

- Further research needed to refine fabrication techniques, explore formulations, and enhance tensile and actuation strength
- More accurate and quantitative tests required for comparing different formulas
- Experimenting with different knot geometries may improve knot tightening and strength

Acknowledgements

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