

LYOTROPIC LIQUID CRYSTAL – BUTYL RUBBER BLENDED NANOMATERIALS

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Report Documentation Page

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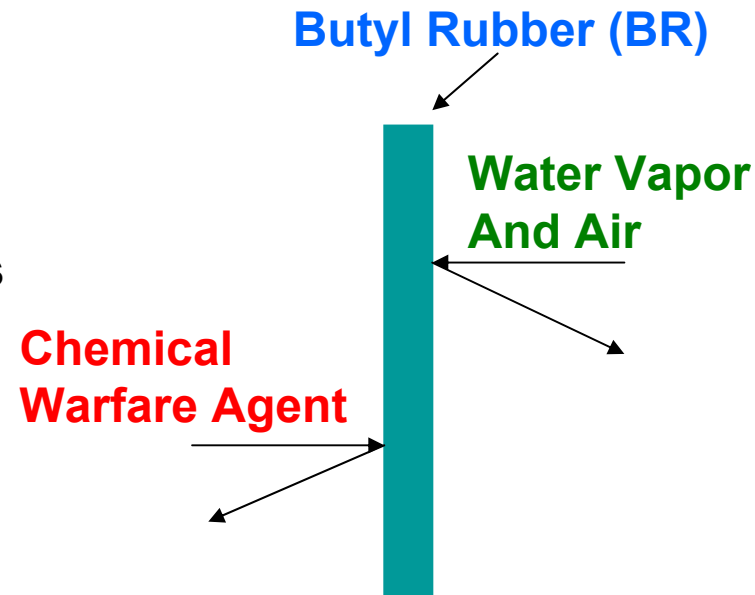
Uses of Butyl Rubber (BR) as barrier material fabric

Advantages:

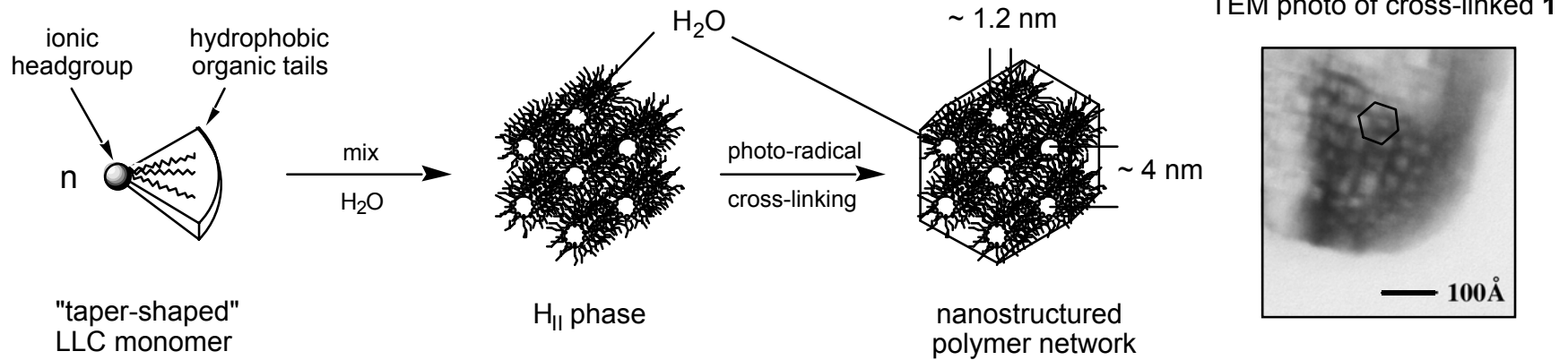
- Low permeability toward gases, organic solvents, water, and reactive chemicals
- Excellent chemical resistance

Disadvantages:

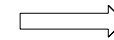
- Lack of permeability of air and water vapor
- ⇒ Development of fatigue and heat stress in wearer



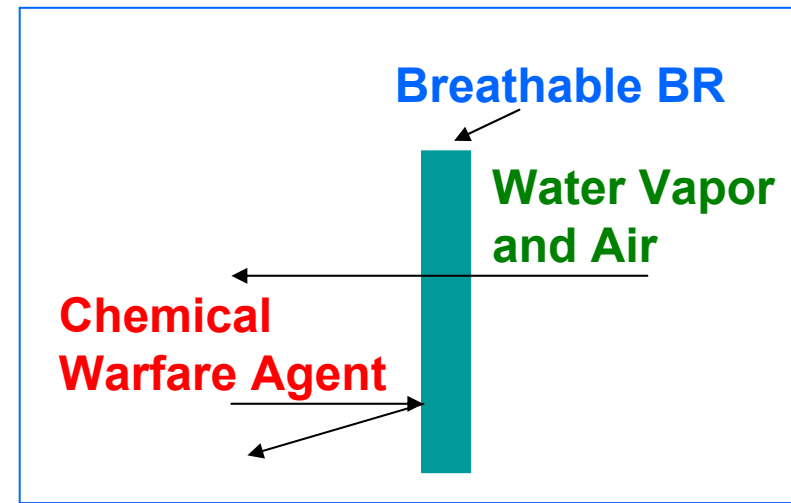
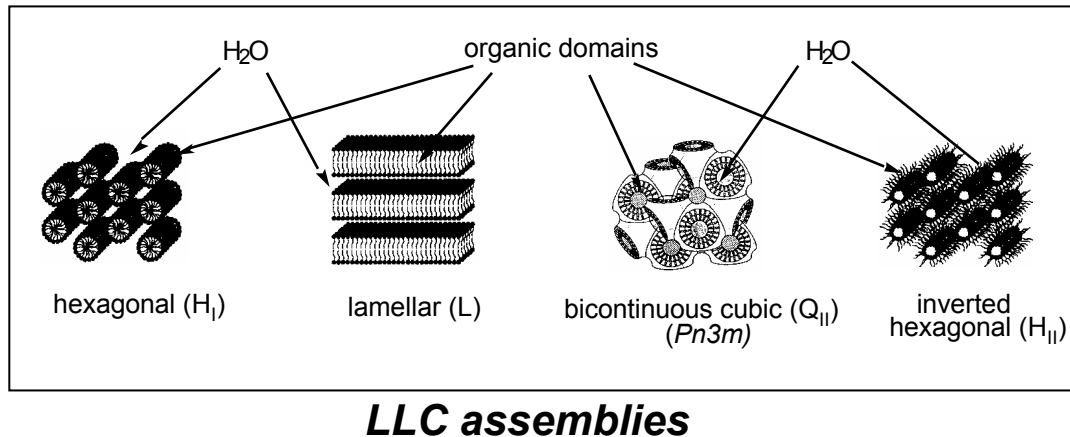
Our Approach



- Blend BR with lyotropic liquid crystals (LLCs) that form inverted hexagonal (H_{II}) phase

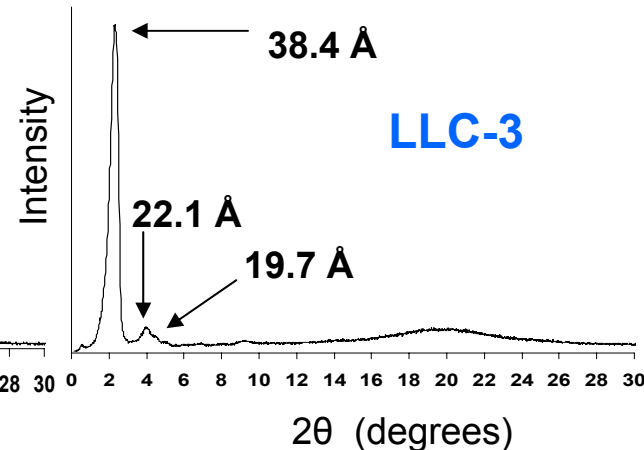
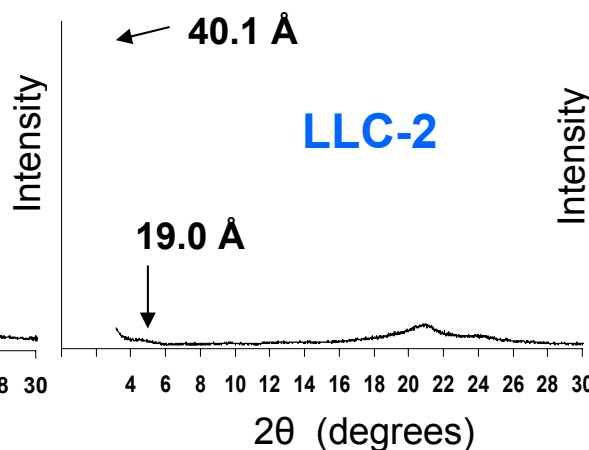
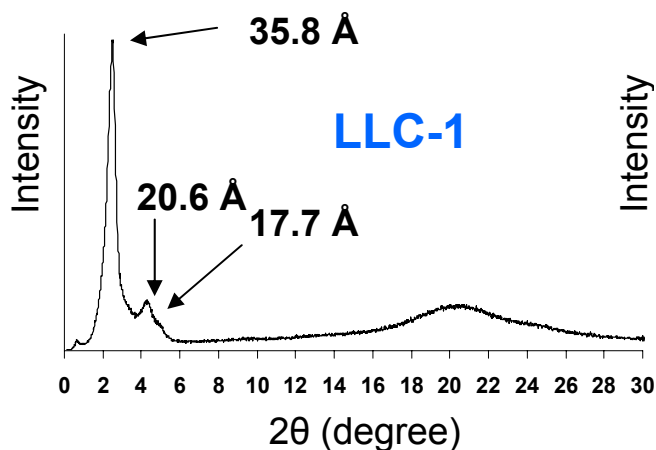
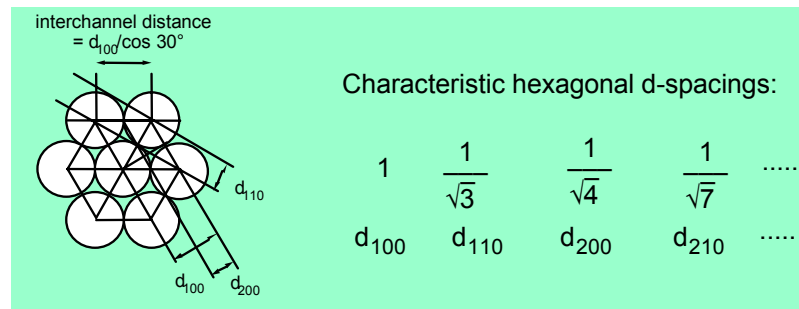
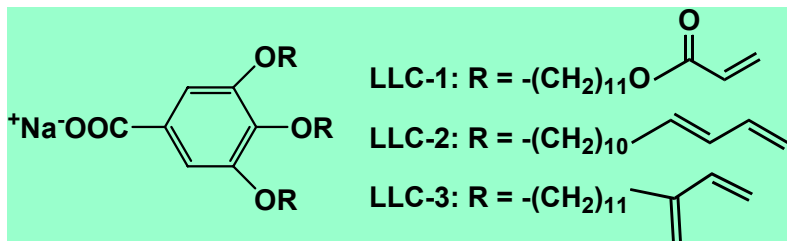


**Nanoporous
Breathable LLC-BR
Composite**



Structure of LLCs and X-ray Diffraction Characterization

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- All three LLCs form H_{II} phase.

Smith, R. C.; Fischer, W. M.; Gin, D. L. *J. Am. Chem. Soc.* 1997, 119, 4092.

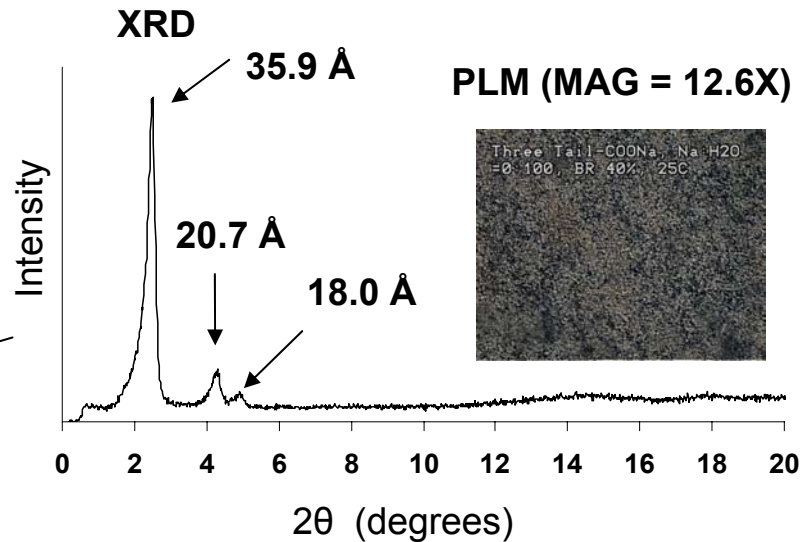
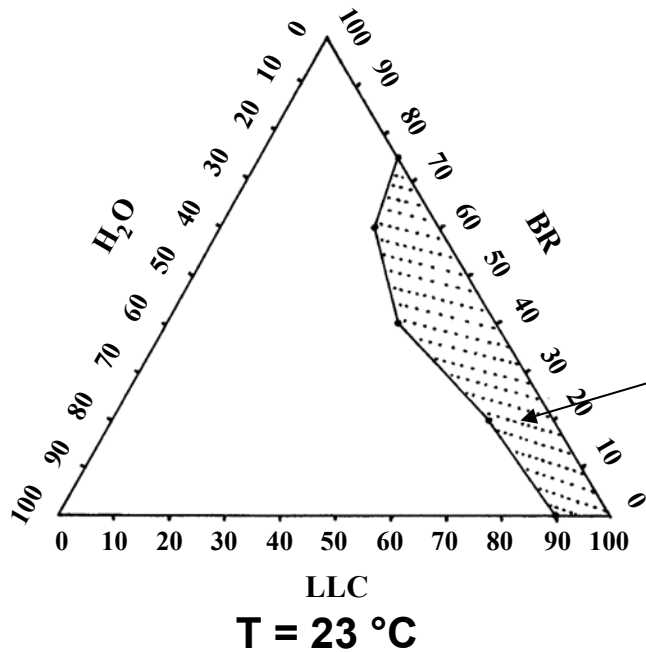
Hoag, B. P.; Gin, D. L. *Macromolecules* 2000, 33, 8549.

Blending Procedure and Structure Determination

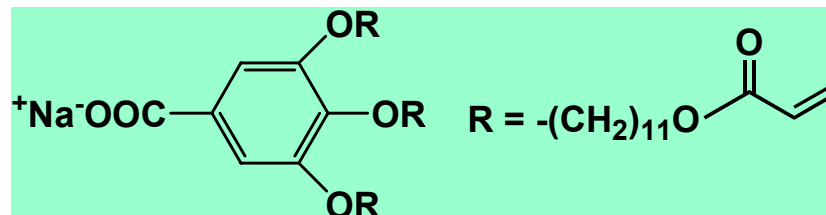
- **LLCs and H₂O were mixed and centrifuged three times (3800 RPM, 15 min.).**
- **Add the LLCs obtained in the above step with BR precursor solution (15 wt % in hexane) and then mix/centrifuge three times (3800 RPM, 15 min.).**
- **Phase structures of samples were determined by X-ray diffraction (XRD) and polarized light microscopy (PLM).**

Phase Diagram of LLC-1 / BR System

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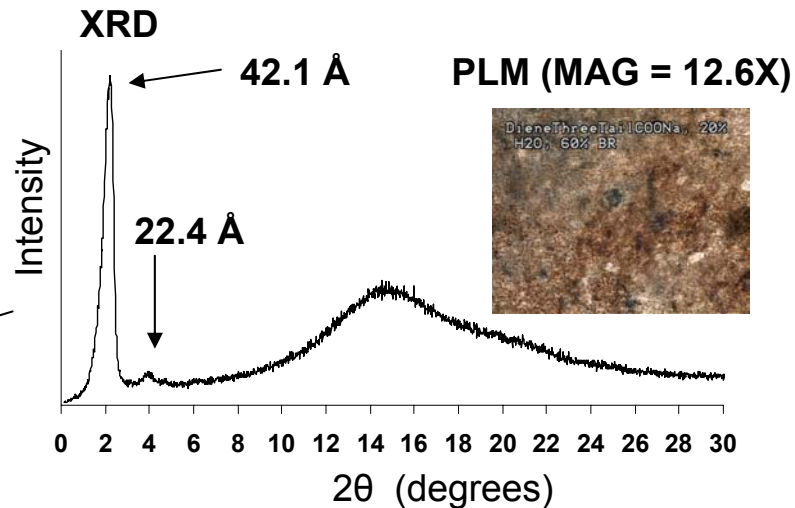
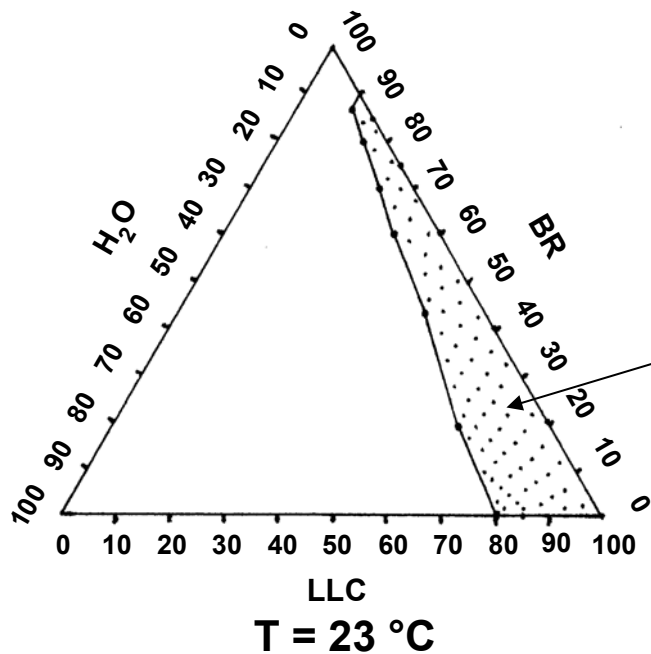
LLC: 60 %; BR: 40 %



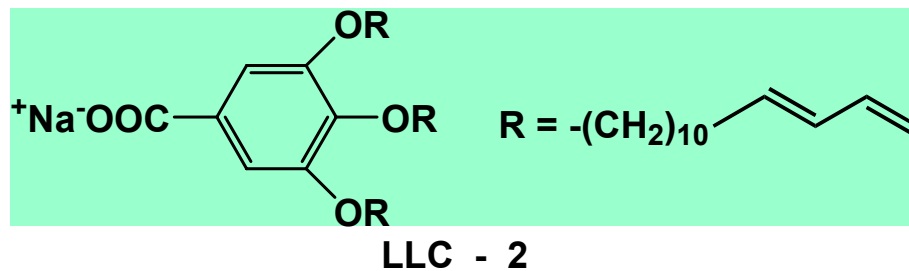
LLC - 1

- LLC-1 retains H_{II} phase even with BR content as high as 75 wt %.
- Retention of H_{II} structure upon photo-initiated radical polymerization.

Phase Diagram of LLC-2 / BR system



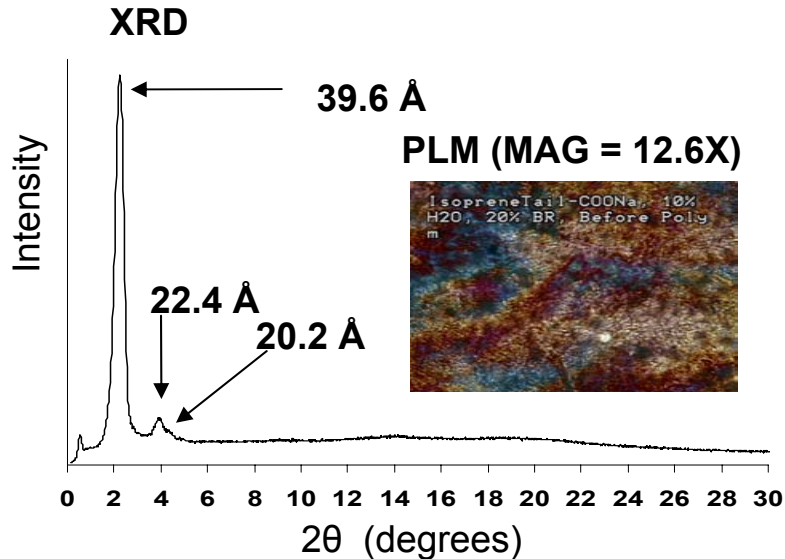
LLC: 32 %; BR: 60 %; H₂O: 8 %



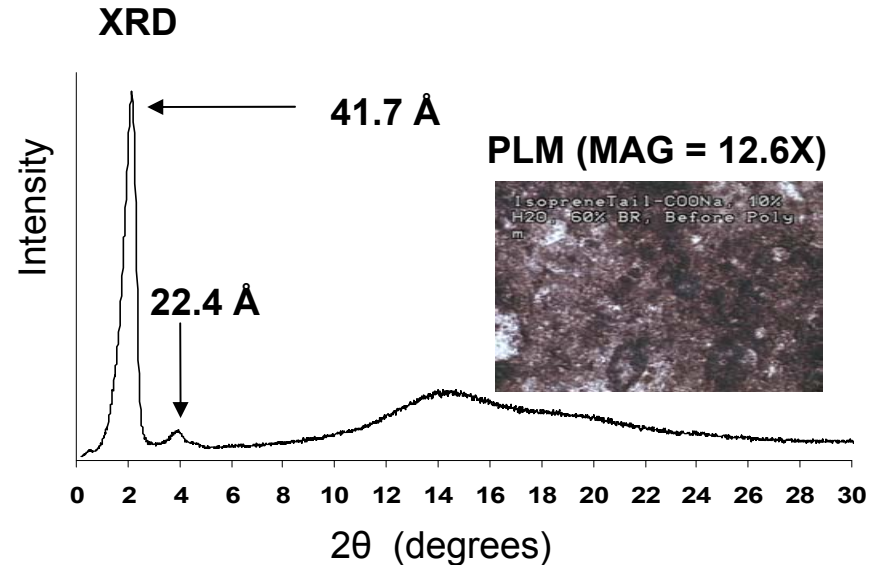
- LLC-2 retains H_∥ phase even with BR content as high as 86 wt %.
- Retention of H_∥ structure upon photo-cross-linking.

XRD Profiles of LLC-3 / BR System

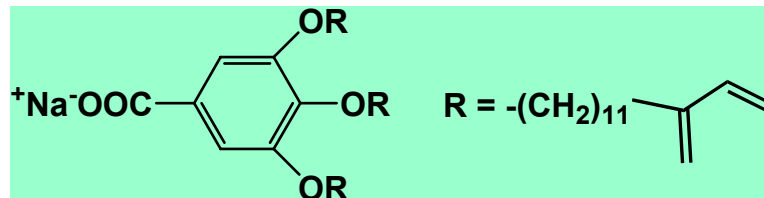
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LLC: 72 %; BR: 20 %; H₂O: 8 %



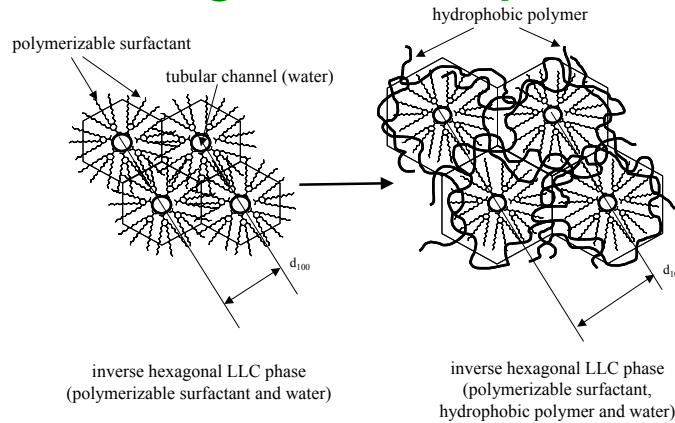
LLC: 36 %; BR: 60 %; H₂O: 4 %



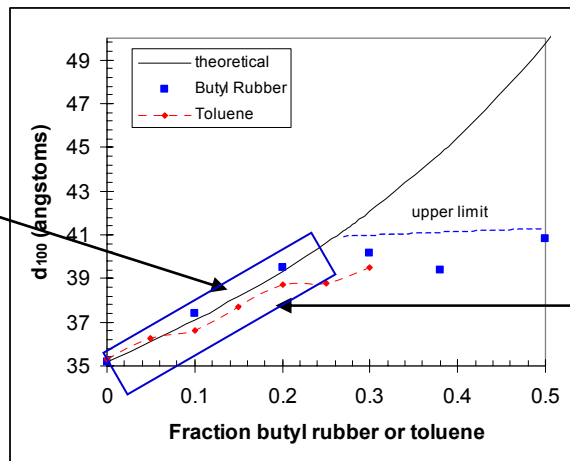
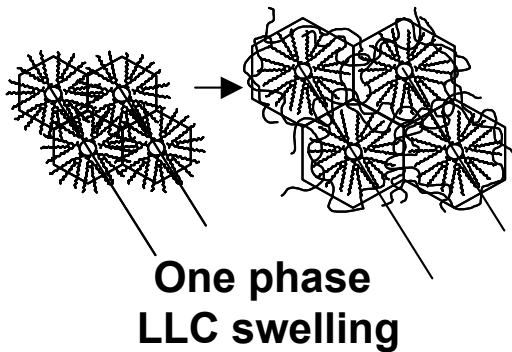
LLC - 3

- LLC-3 retains H_{II} phase after mixing with up to 70 wt % BR.
- Retention of H_{II} structure upon photopolymerization.

Swelling of the LLC phase



Experimental vs. theoretical swelling:
 Both toluene and butyl rubber can swell the theoretical maximum up to about 25%



PLM (MAG = 12.6X)

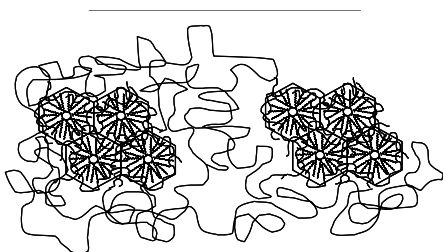
Pure LLC

20 % BR

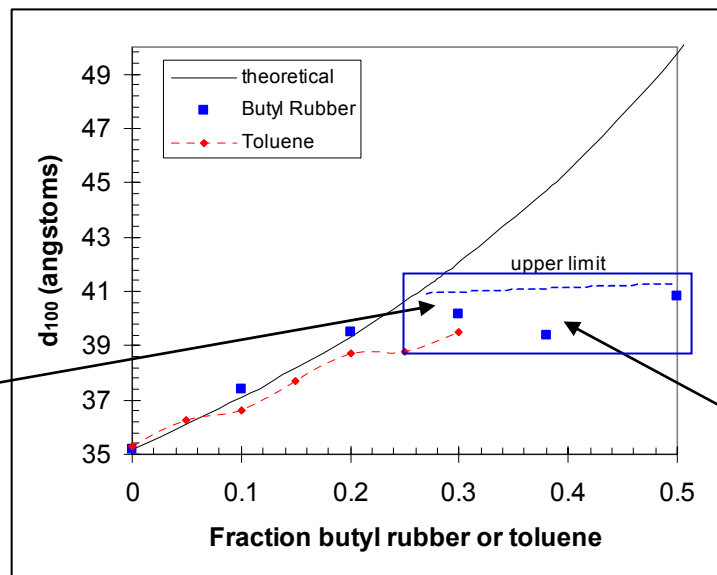


- Unit cell of LLC phase increases with increasing BR content, up to 25 wt %.

Experimental vs. theoretical swelling:
Both toluene and butyl rubber can swell the theoretical maximum up to about 25%



**Two phase
LLC swelling**



PLM (MAG = 12.6X)

50 % BR



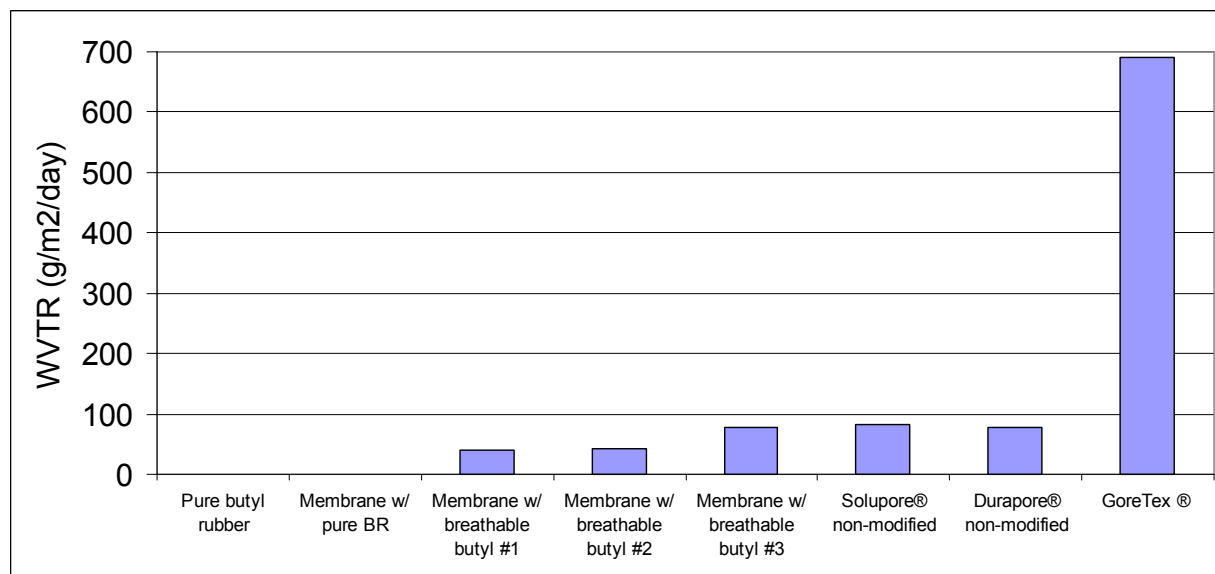
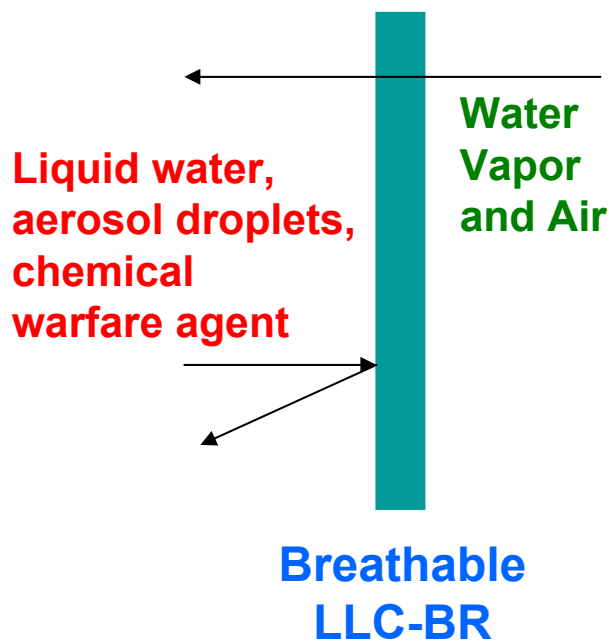
75 % BR



- Above 25 wt % BR, BR chains go between LLC domains, and the phase separation occurs.**

Breathable BR membranes were produced using an ultraporous membrane support. The ultimate goal is to allow water vapor transport, while preventing chemical warfare agent (CWA) penetration.

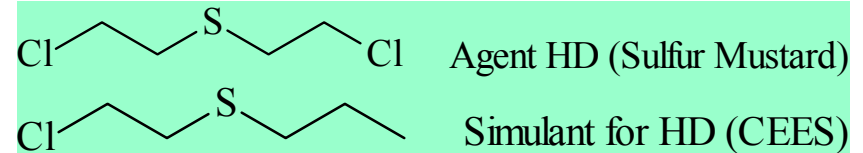
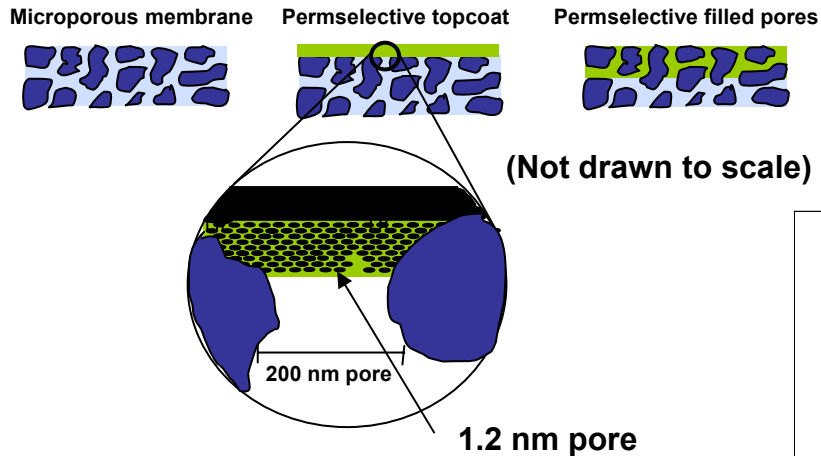
- Water vapor transmission rate (WVTR) through membranes.
- 25 °C, 80% relative humidity to 0%.
- Mass transfer limited by ultraporous membrane support.
- Breathable BR composite membranes have 10% of the WVTR (“breathability”) of GoreTex®



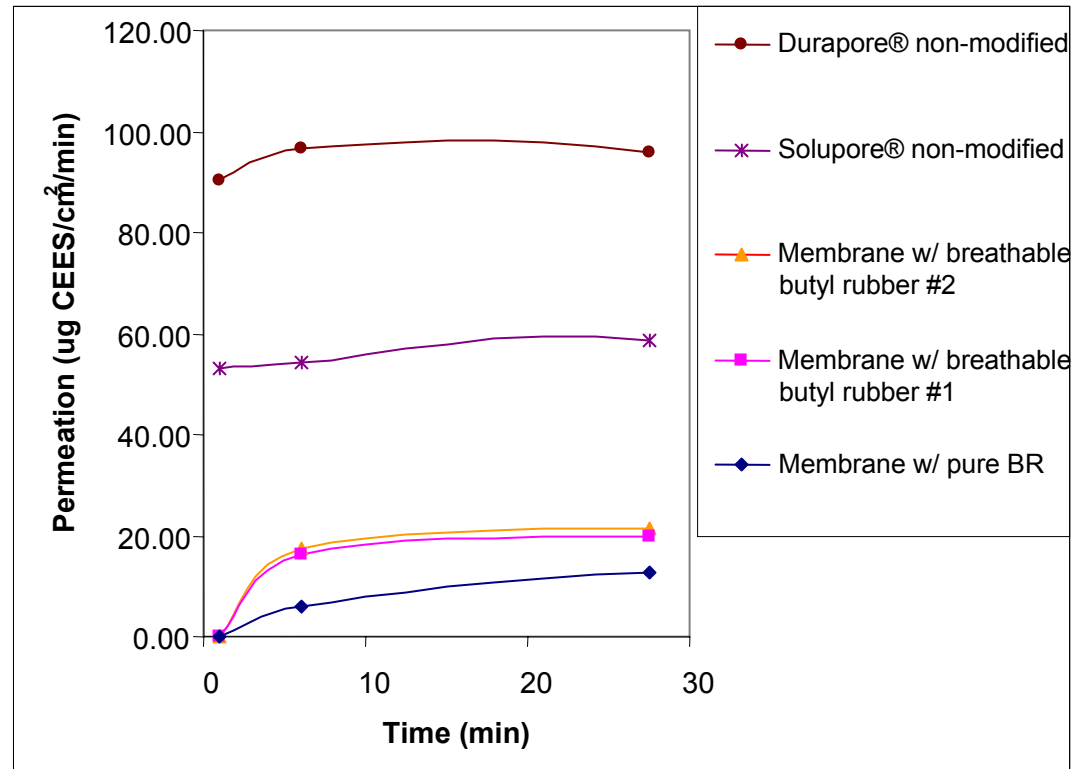
Note: GoreTex® has a higher reported WVTR for other testing conditions. The results above are for comparison purposes only.

Chemical Warfare Agent Simulant Permeation Through Breathable LLC - BR Nanocomposites

Composite membranes



- Composite membranes were prepared with LLC / BR composites with varying amounts of BR.
- CEES permeation is compared for un-modified membrane supports and a composite membrane with either a breathable BR or a pure BR top-coat.
- Note that if the membranes were defect free, the pure BR should exhibit a higher resistance to CEES permeation than observed.
- Therefore, we expect even lower permeation for LLC / BR composites (breathable BR) with improved membrane production techniques.



Feed conditions: saturated CEES vapor, STP
Sweep conditions: N₂ and water vapor (25% rel. hum. At 25°C)

Summary

- Nanoporous polymer composites were prepared by blending and copolymerizing LLC monomers with commercial BR polymer.
- The LLCs in the resulting polymer composites form the H_{II} phase and allow air and water vapor to permeate, but retard permeation of chemical agent simulants.

Future Work

- More detailed investigations on the vapor permeation of the LLC-BR composites are in progress.

Acknowledgments

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