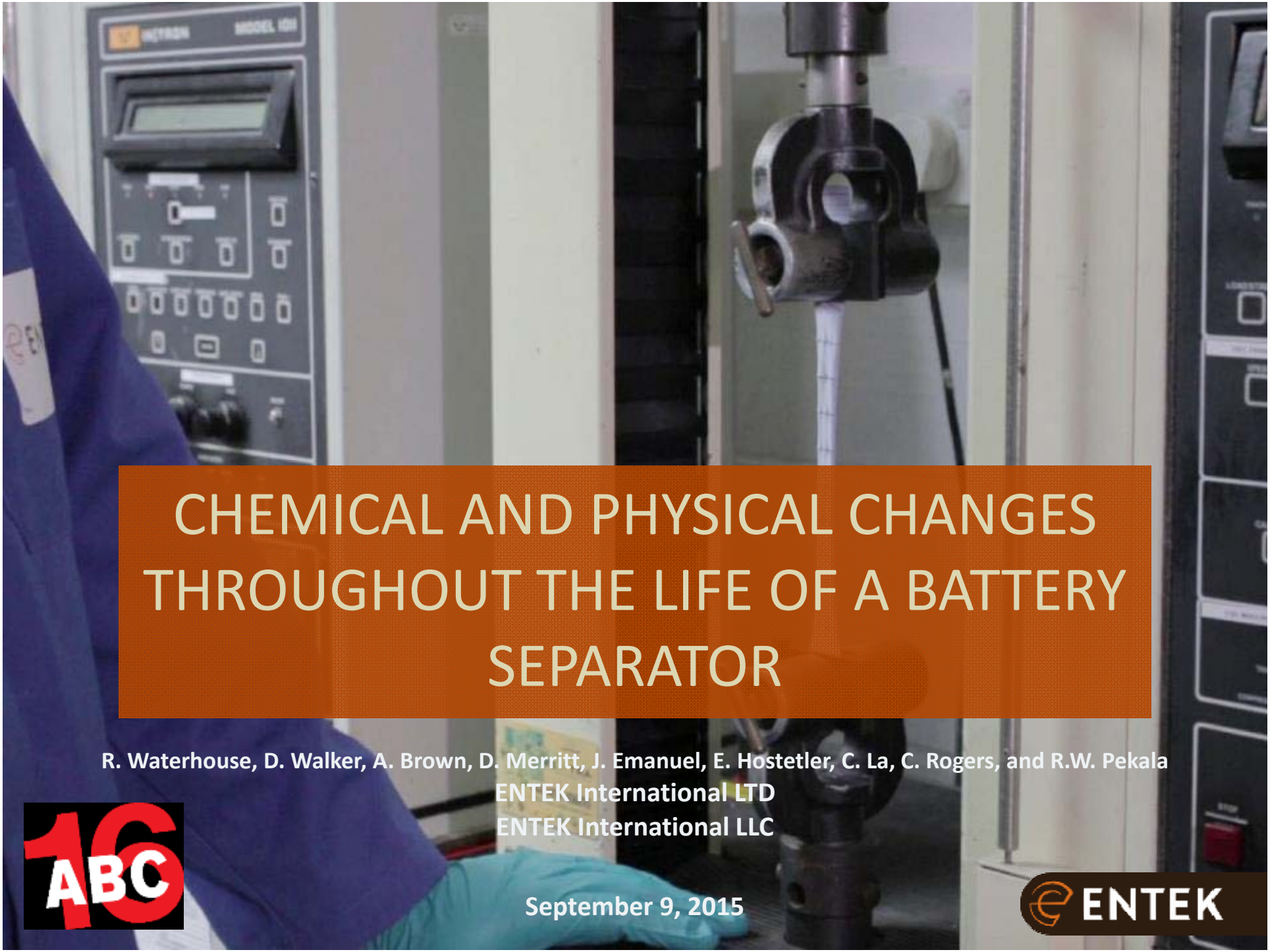
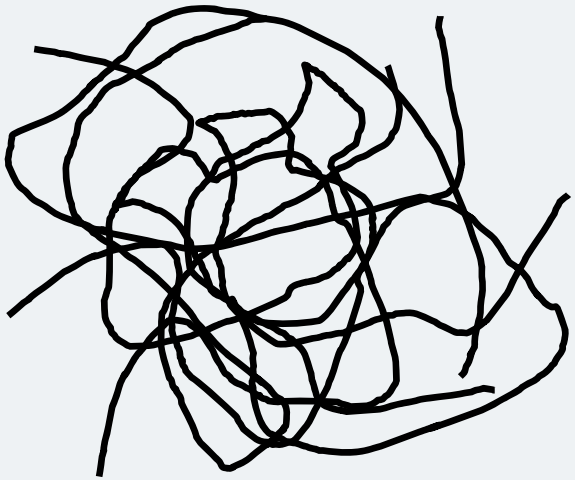


CHEMICAL AND PHYSICAL CHANGES THROUGHOUT THE LIFE OF A BATTERY SEPARATOR

R. Waterhouse, D. Walker, A. Brown, D. Merritt, J. Emanuel, E. Hostetler, C. La, C. Rogers, and R.W. Pekala
ENTEK International LTD
ENTEK International LLC

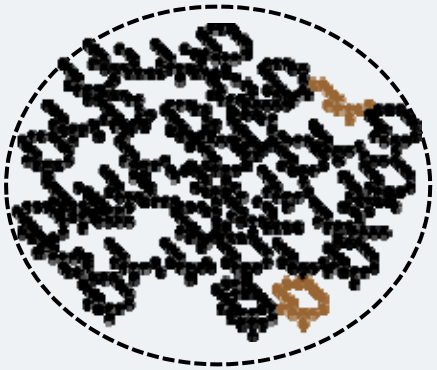
September 9, 2015

UHMWPE GEL PROCESSING



UHMWPE

$(-CH_2-CH_2-)_x$



Silica

Oil

Twin Screw

Extrusion

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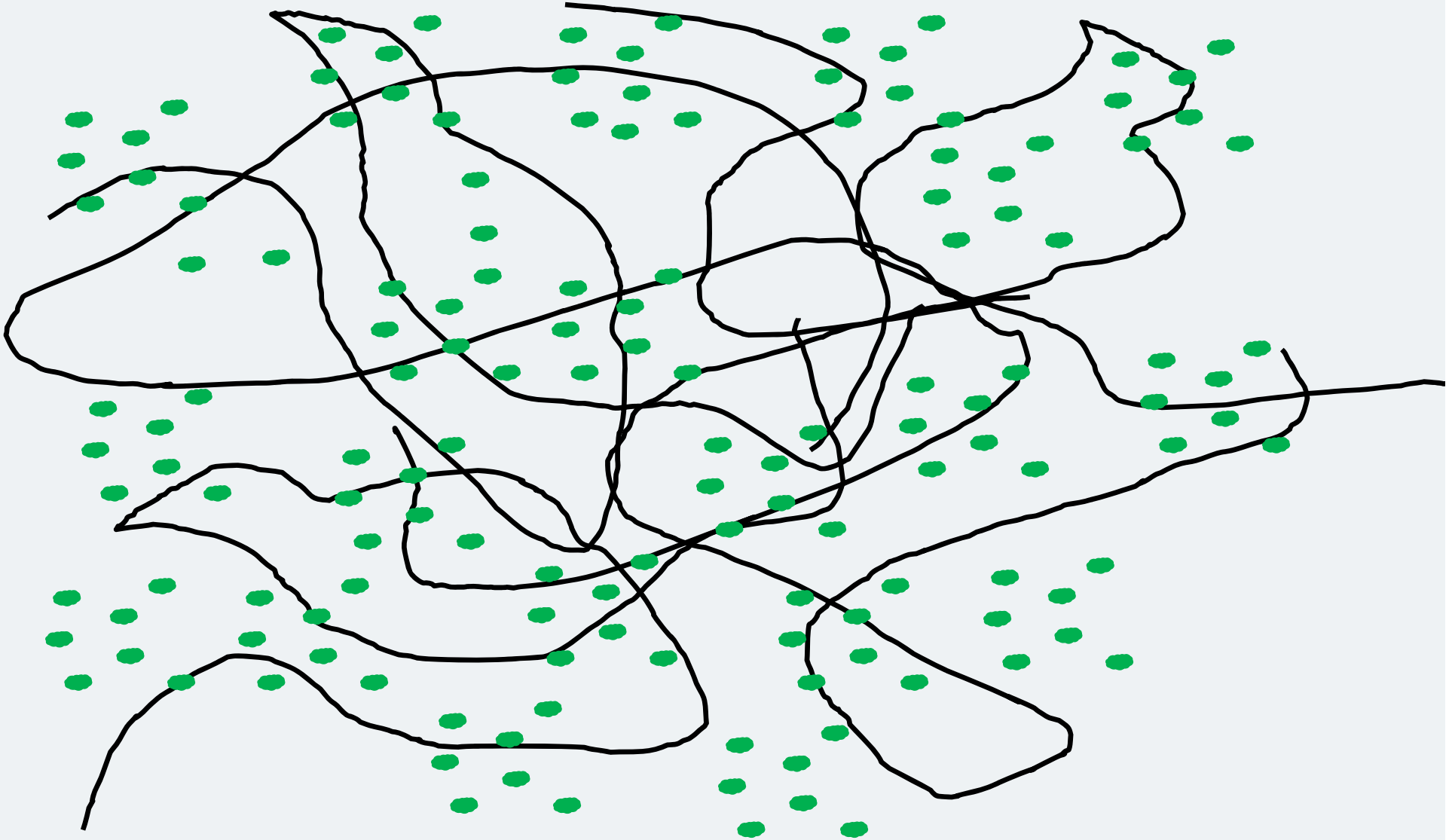
UHMWPE + OIL + SHEAR → CHAIN DISENTANGLEMENT



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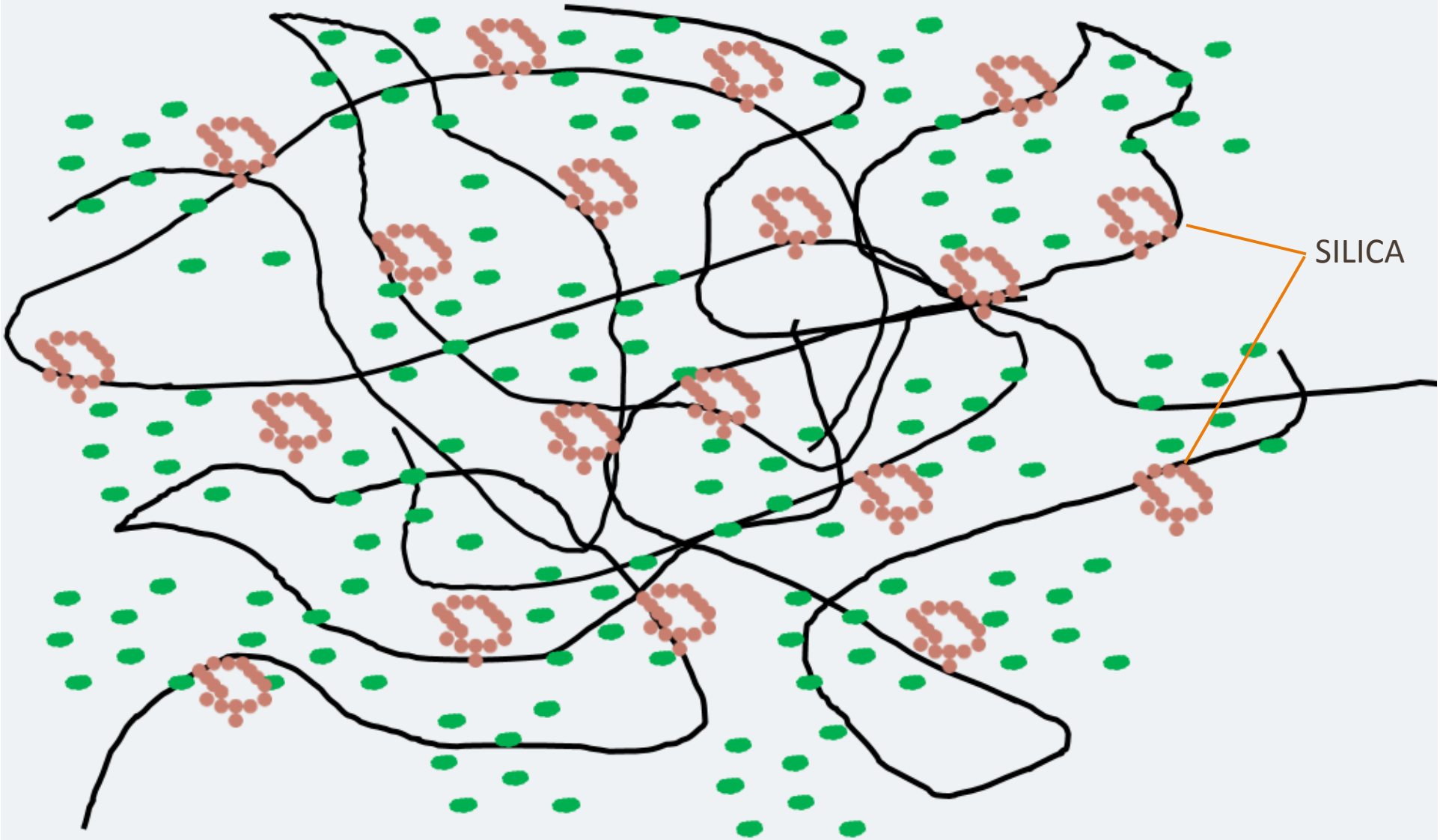
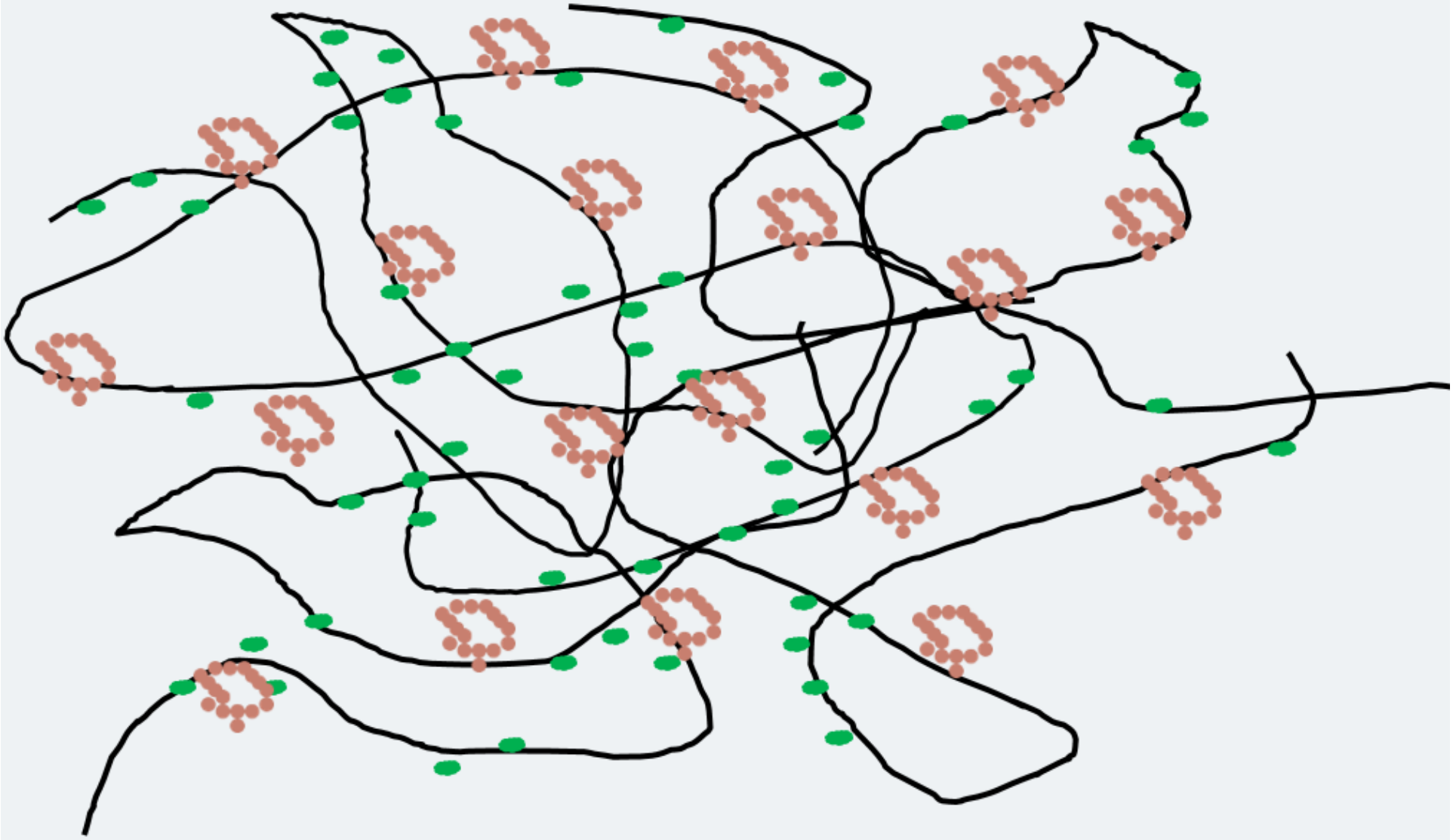
SILICA AGGREGATES DISPERSED THROUGHOUT THE POLYMER MATRIX



OIL EXTRACTION + SOLVENT DRYING → POROUS SEPARATOR



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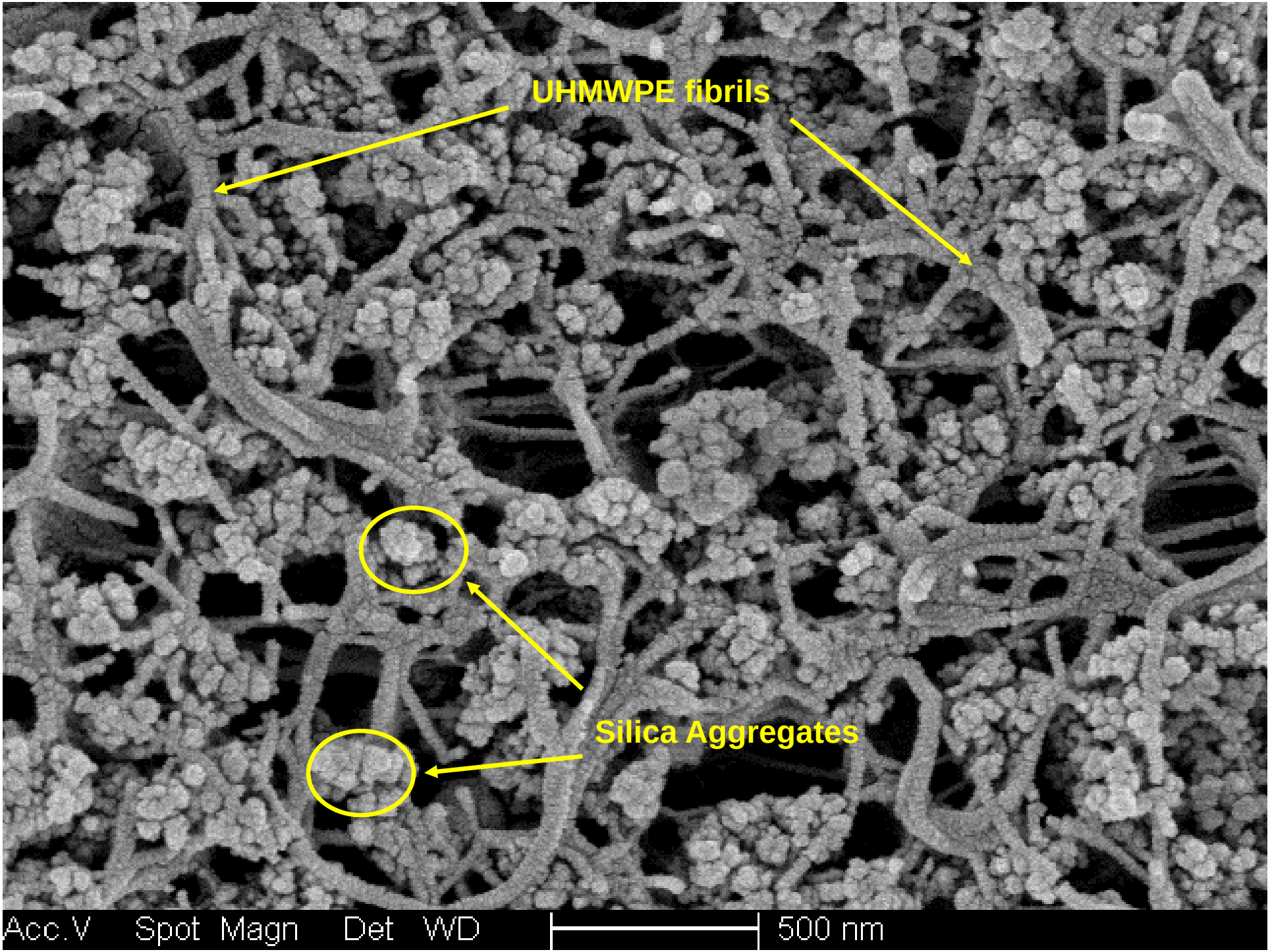
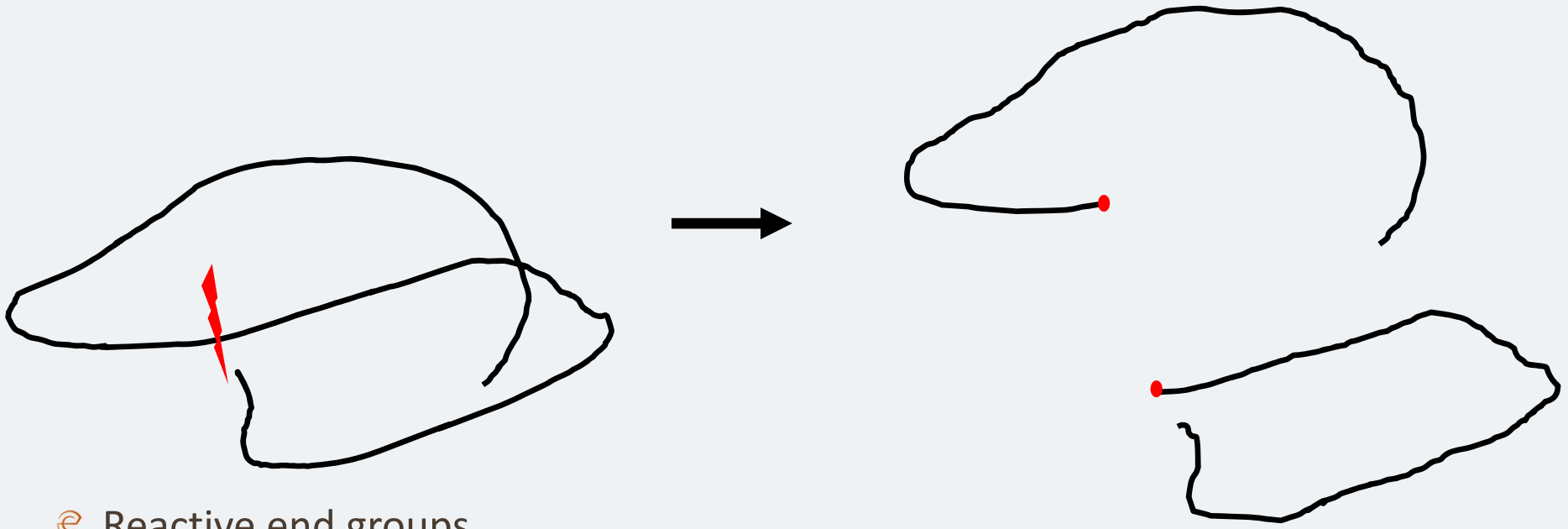

UHMWPE fibrils
Silica Aggregates
Acc.V Spot Magn Det WD
500 nm

DEGRADATION MECHANISM - SHEAR

- UHMWPE chains are susceptible to chain scission during twin-screw extrusion

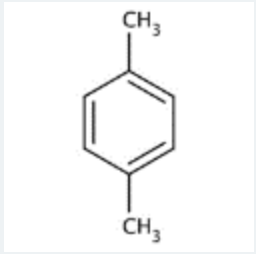


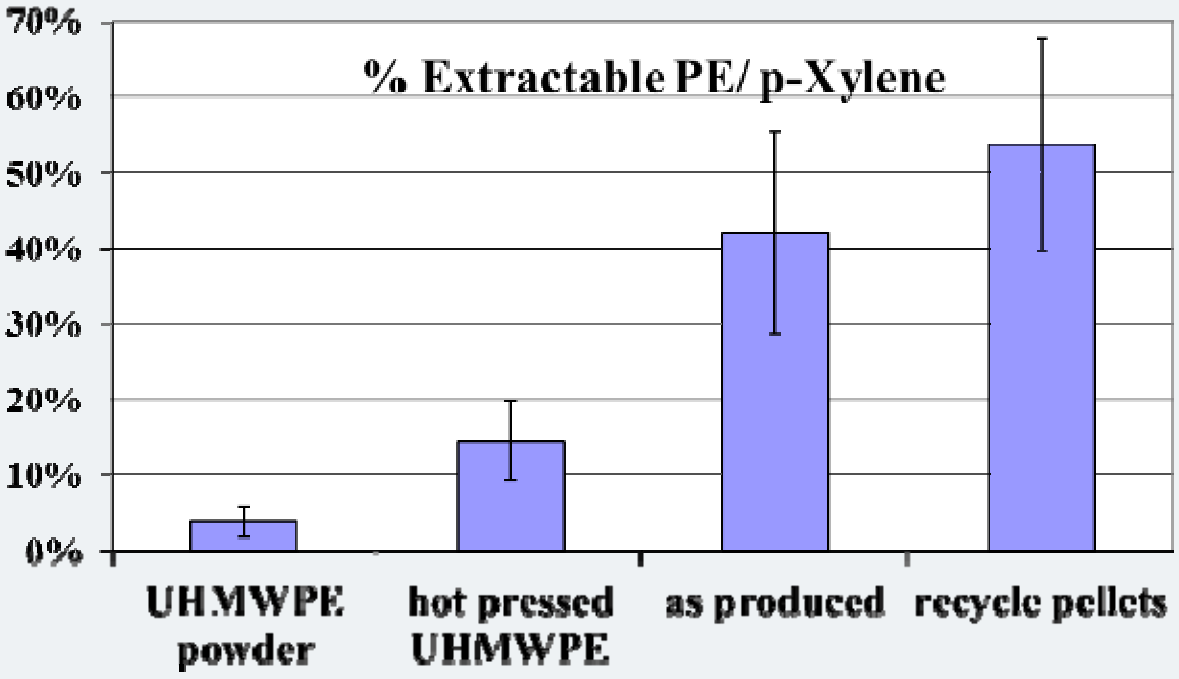
- Reactive end groups
  - Hydrogen abstraction
  - Oxidation
  - Crosslinking

$$R\bullet + O_2 \rightarrow ROO\bullet$$

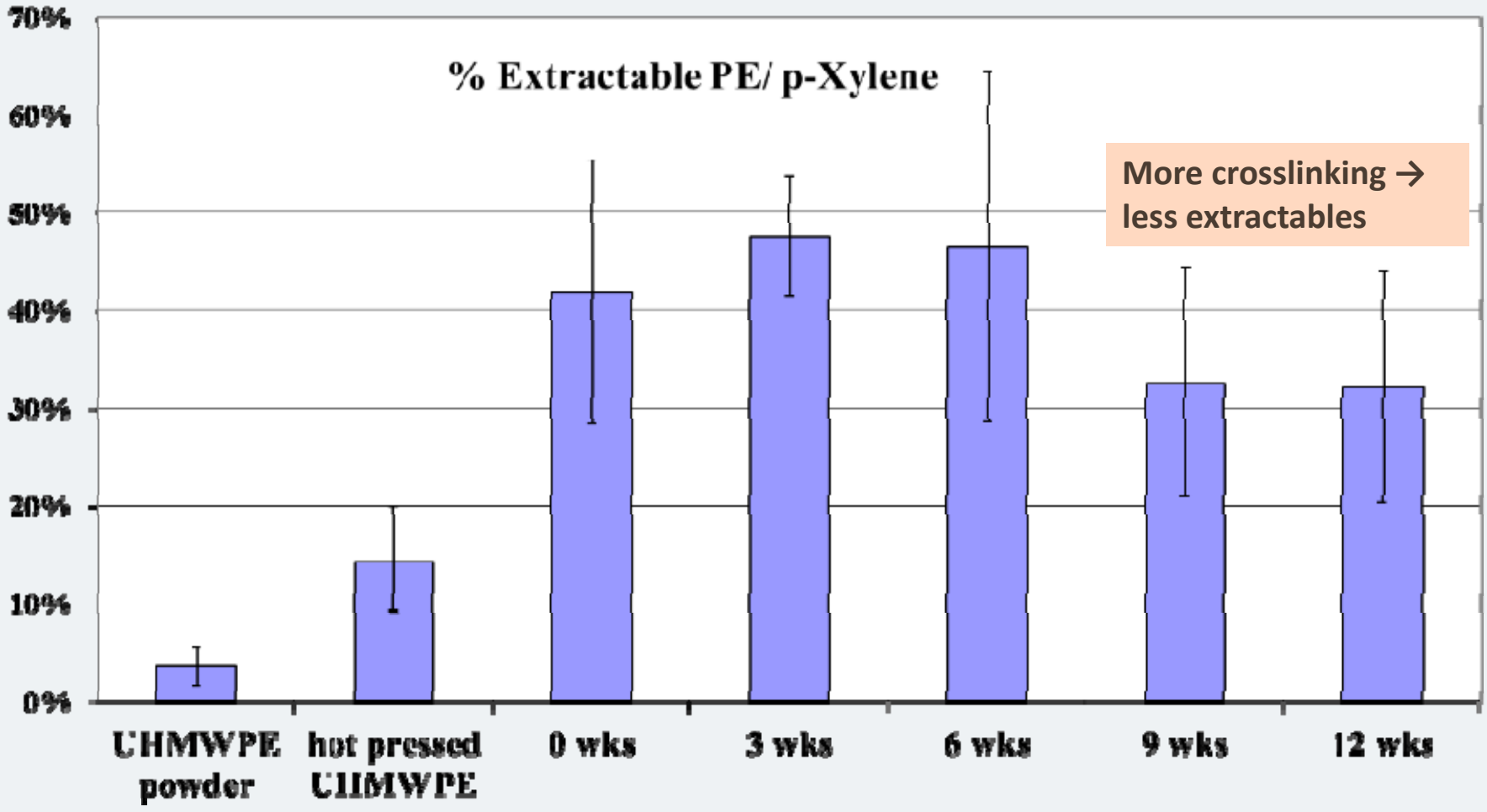
EVIDENCE OF SHEAR DEGRADATION

- Soxhlet extraction
  - Para-xylene
  - 138°C



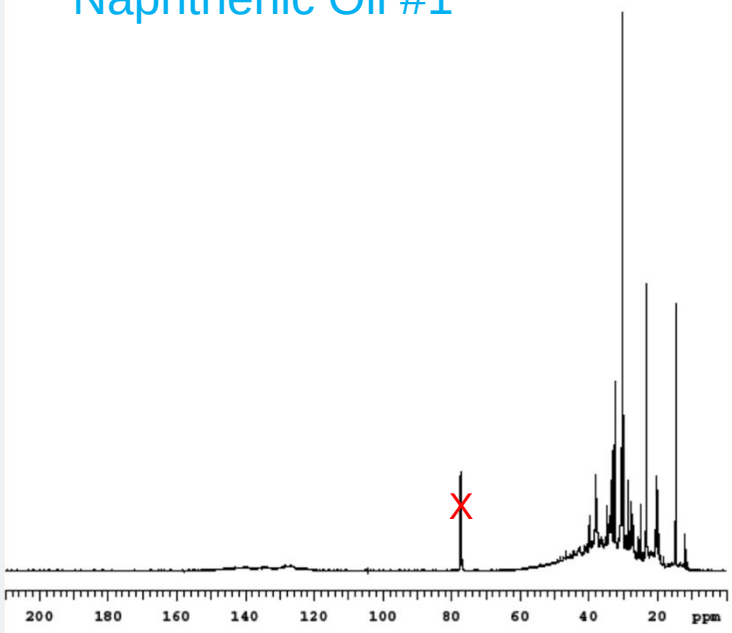


CHAIN SCISSION VS. CROSSLINKING IN BATTERY ENVIRONMENT

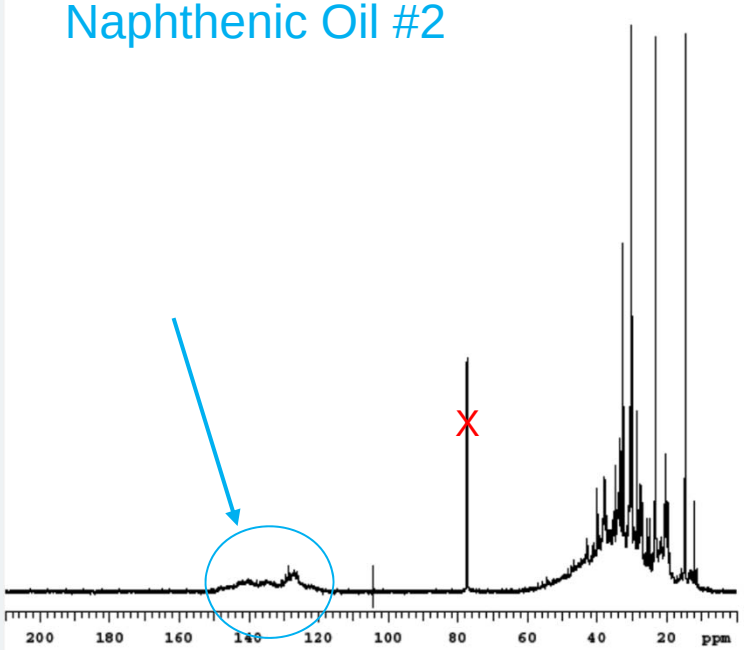


PROCESS OILS ARE COMPLEX CHEMICAL MIXTURES

Naphthenic Oil #1



Naphthenic Oil #2



C-13 NMR Analysis provides a chemical fingerprint for process oils

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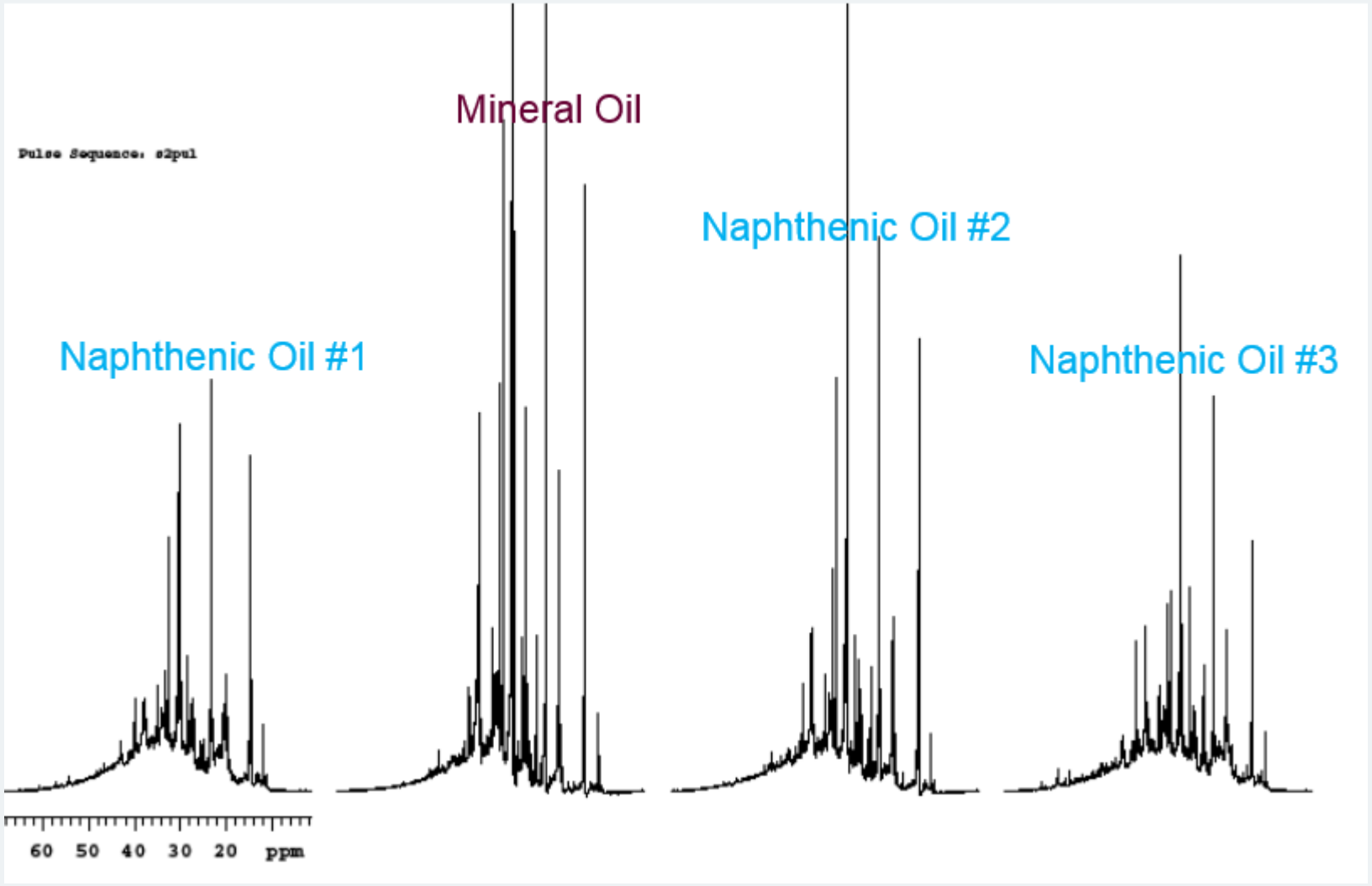
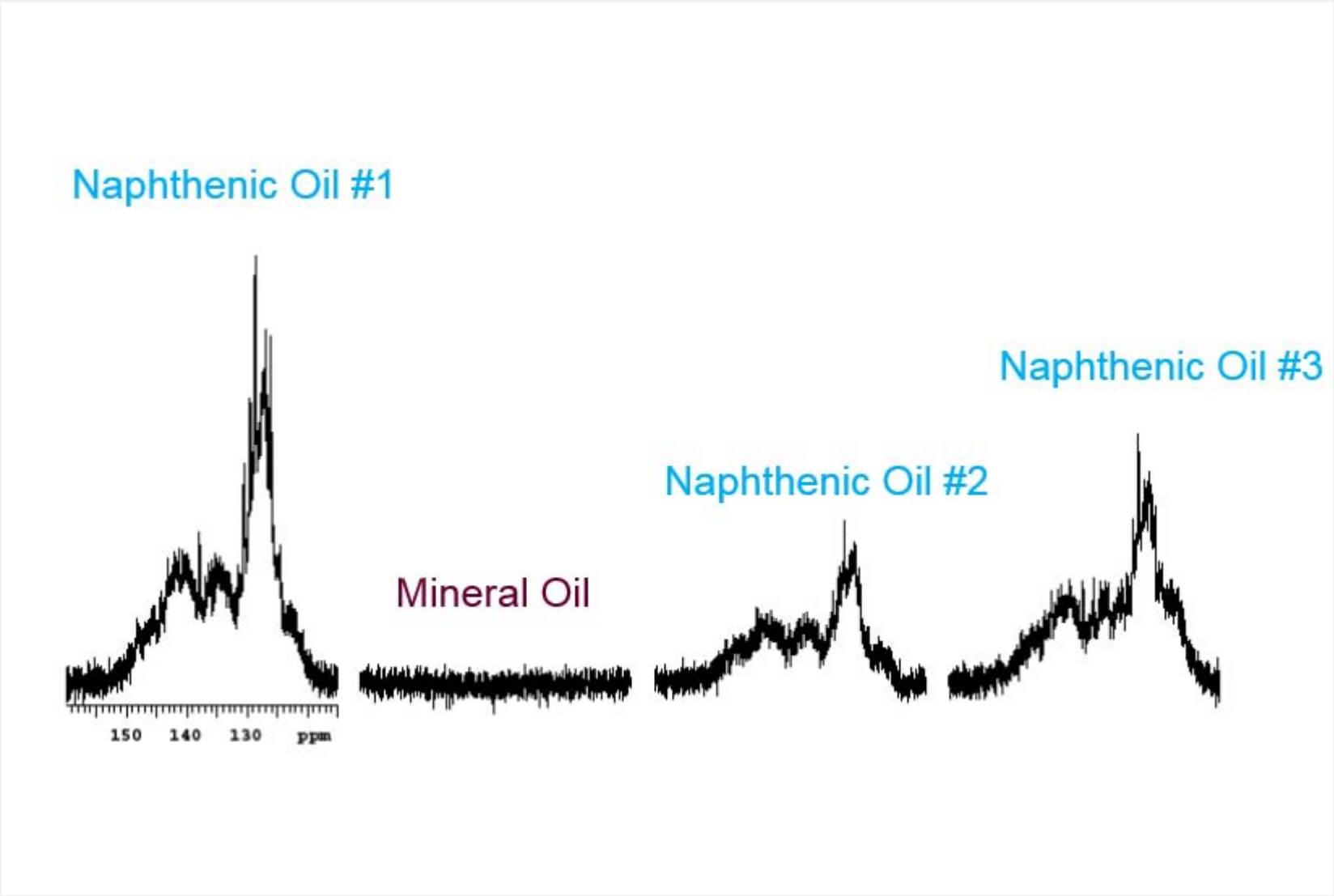
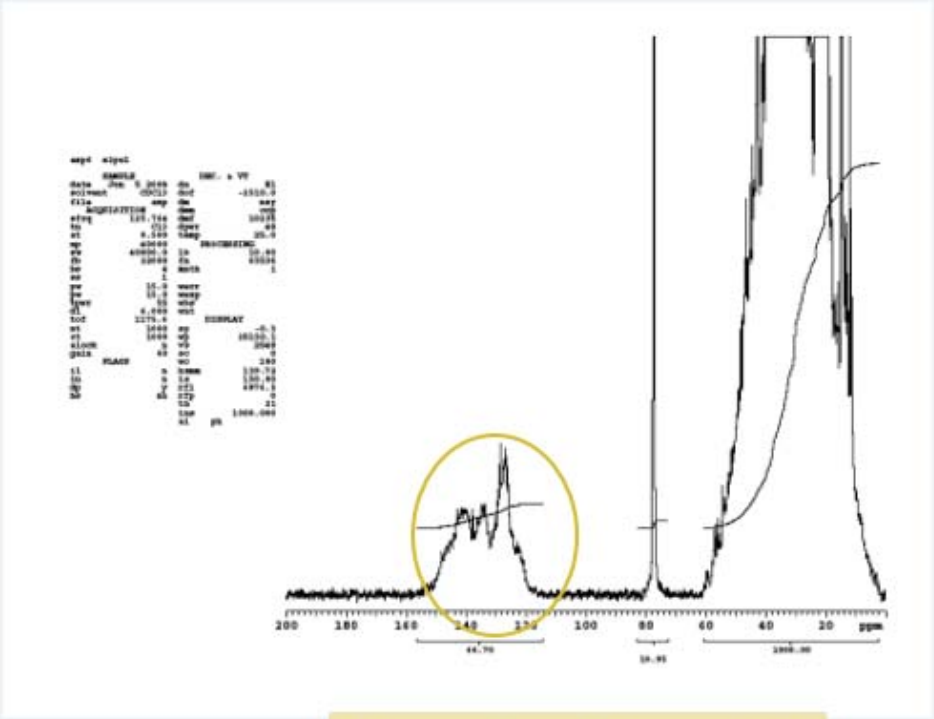
C-13 NMR ANALYSIS (ALIPHATICS)



C-13 NMR ANALYSIS (AROMATICS)

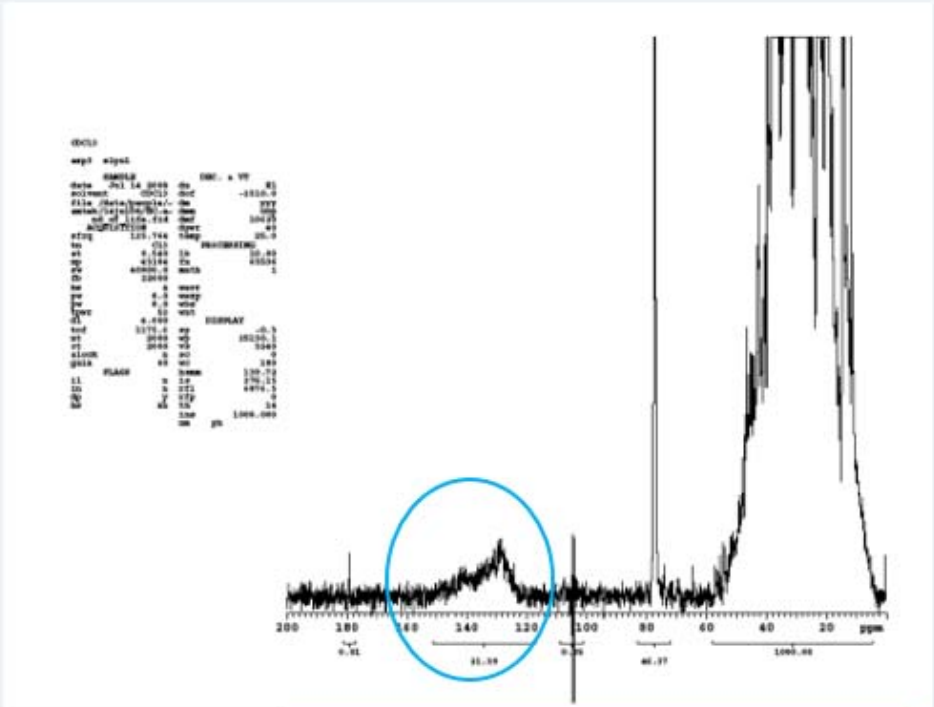


PROCESS OIL --- CHEMICAL CHANGES IN BATTERY ENVIRONMENT



Virgin Naphthenic Oil

Area = 67 units



Naphthenic Oil extracted after battery failure

Area = 21 units

SEPARATOR OXIDATION RESISTANCE

- Test methods
  - Perox 80
  - Potassium dichromate
  - Oxidation induction time
  - Electrochemical oxidation test
  - High temperature battery life test

- Material considerations
  - Polymer matrix
  - Residual oil
  - Oil / PE ratio

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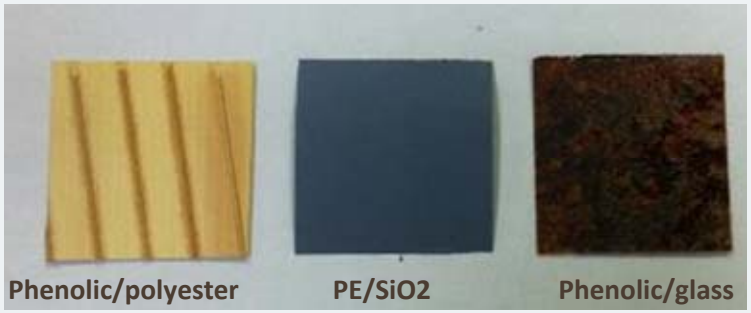
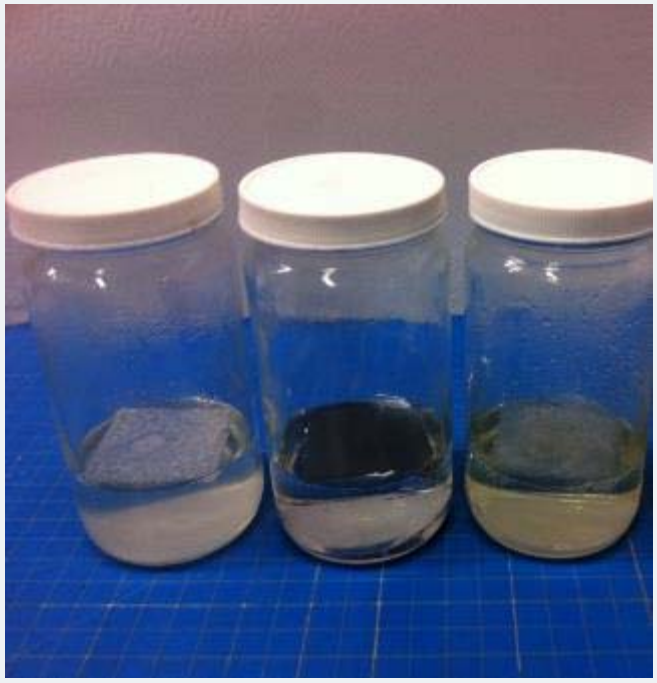
CHEMICAL RESISTANCE VS. OXIDATION RESISTANCE

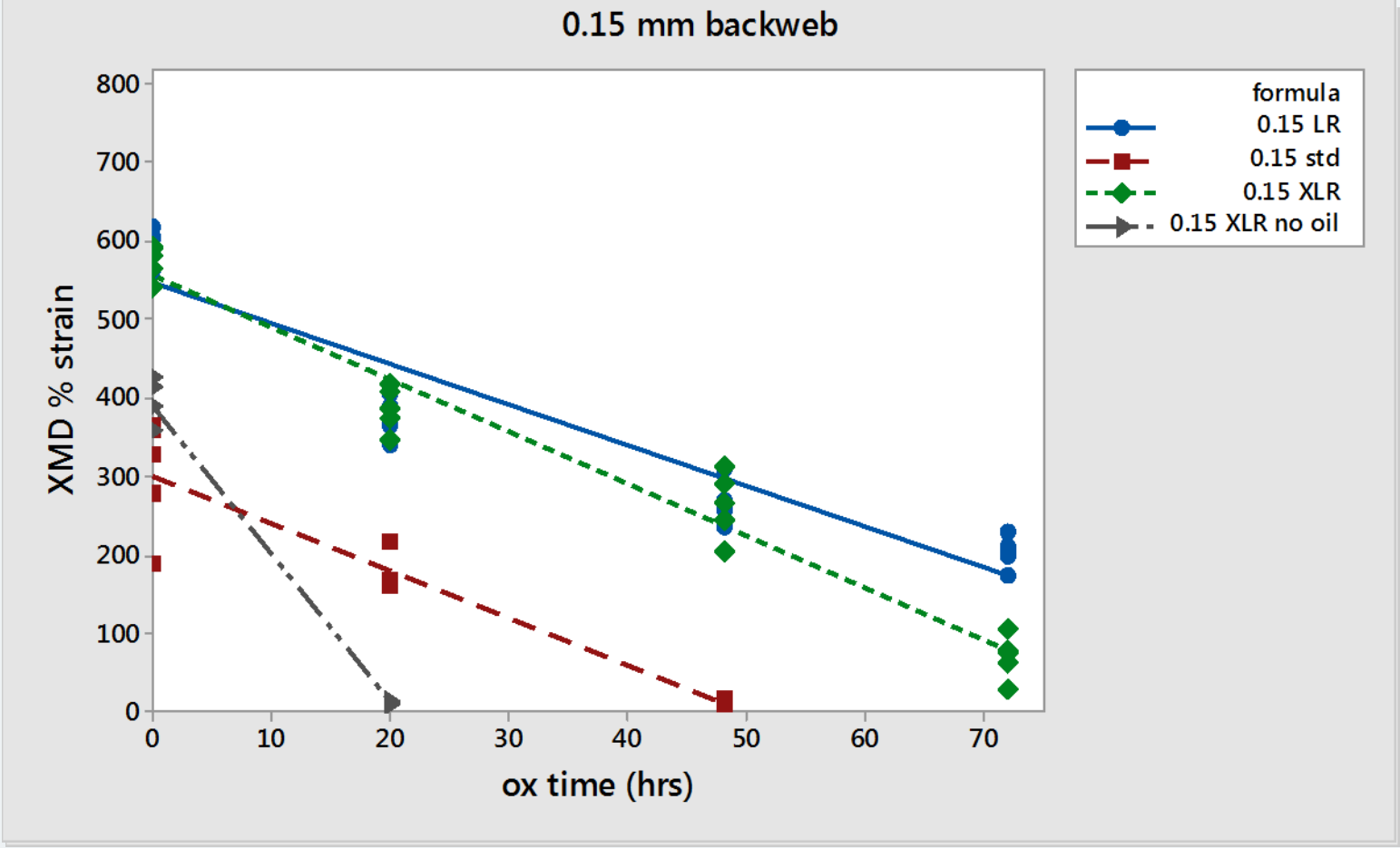


After 20 hrs in H_2SO_4 at 80 °C



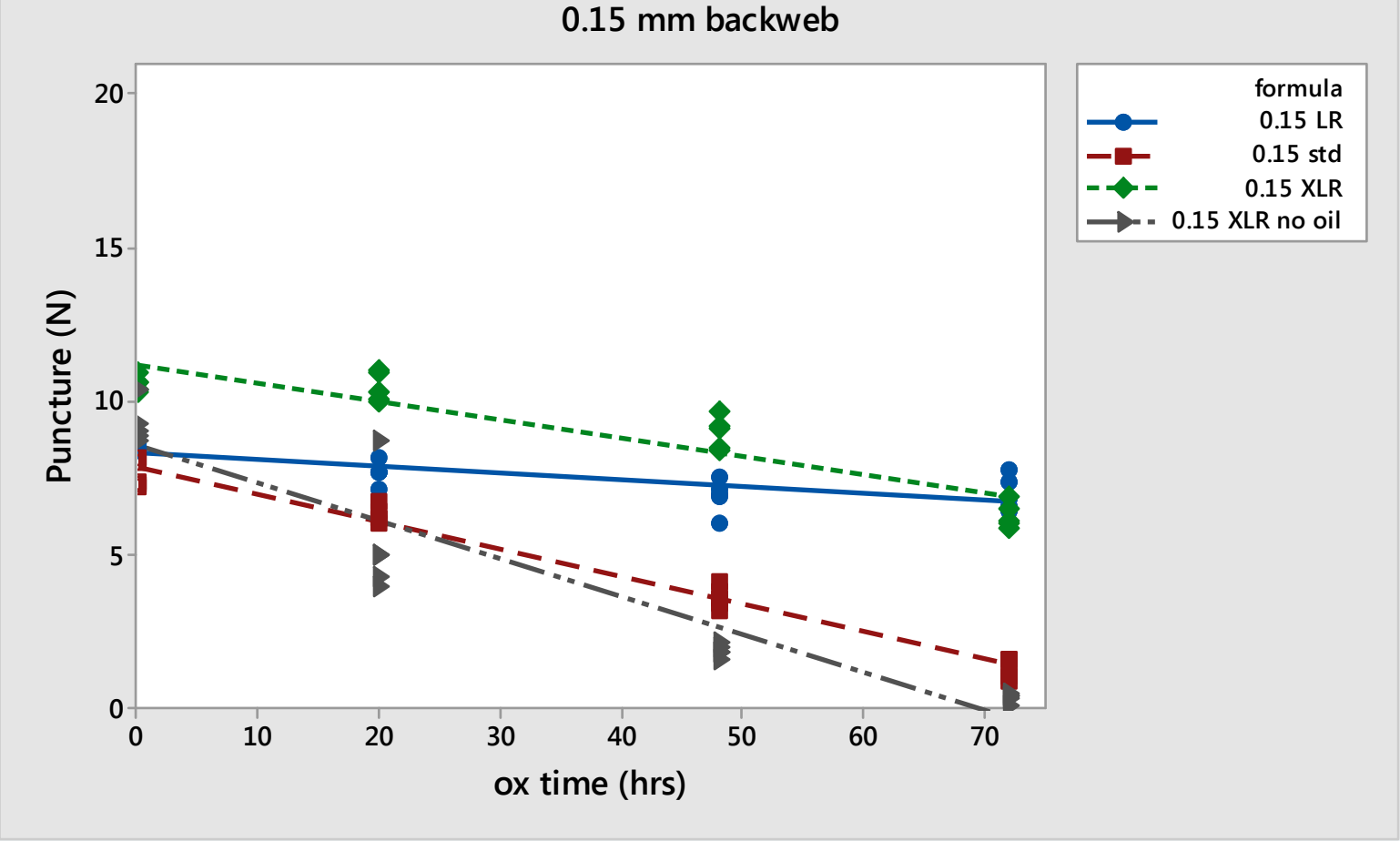
After 20 hrs in H_2SO_4/H_2O_2 at 80 °C

PEROX 80 --- % XMD ELONGATION VS EXPOSURE TIME



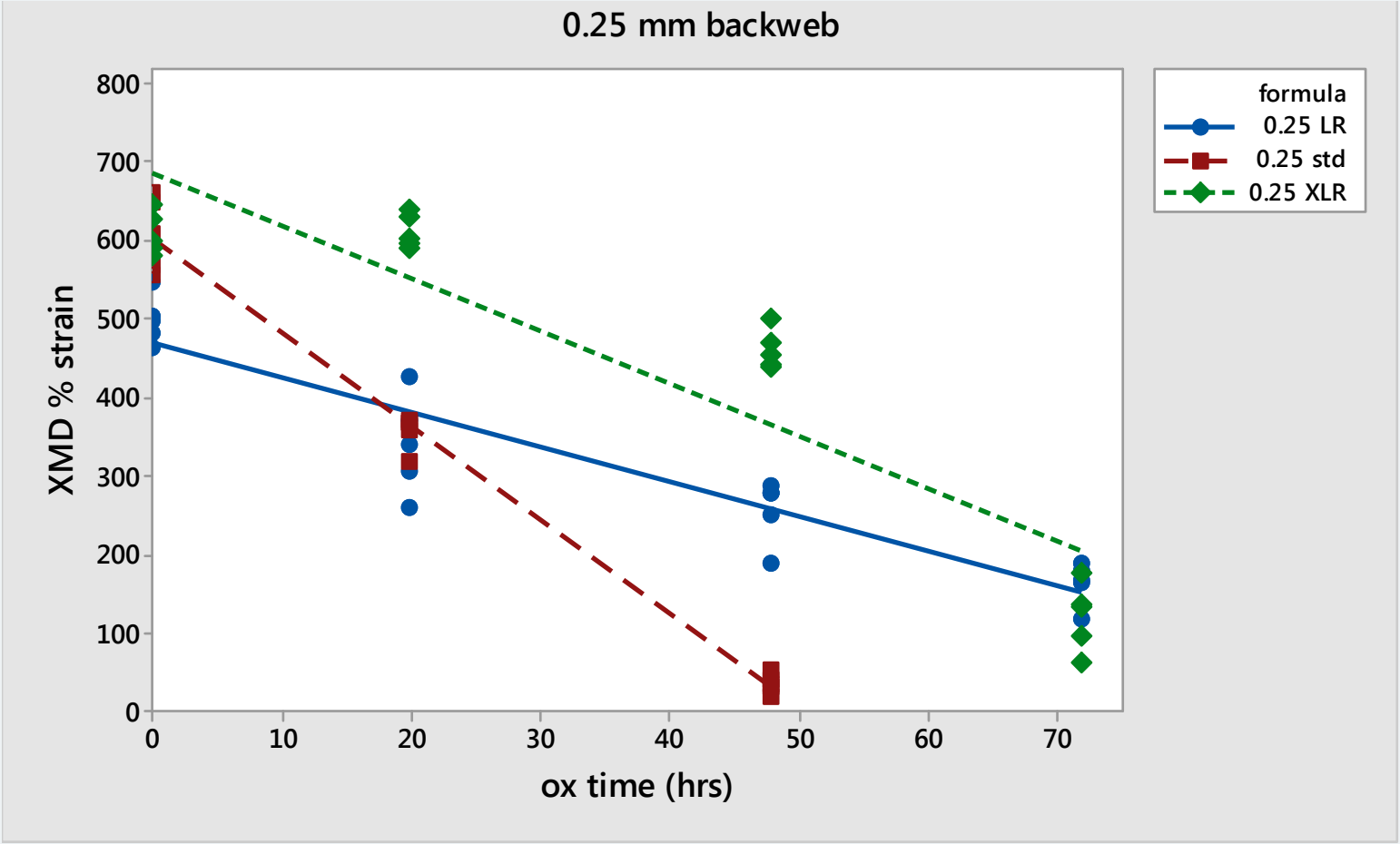
Polymer content : XLR > LR > STD

PEROX 80 --- PUNCTURE STRENGTH VS EXPOSURE TIME



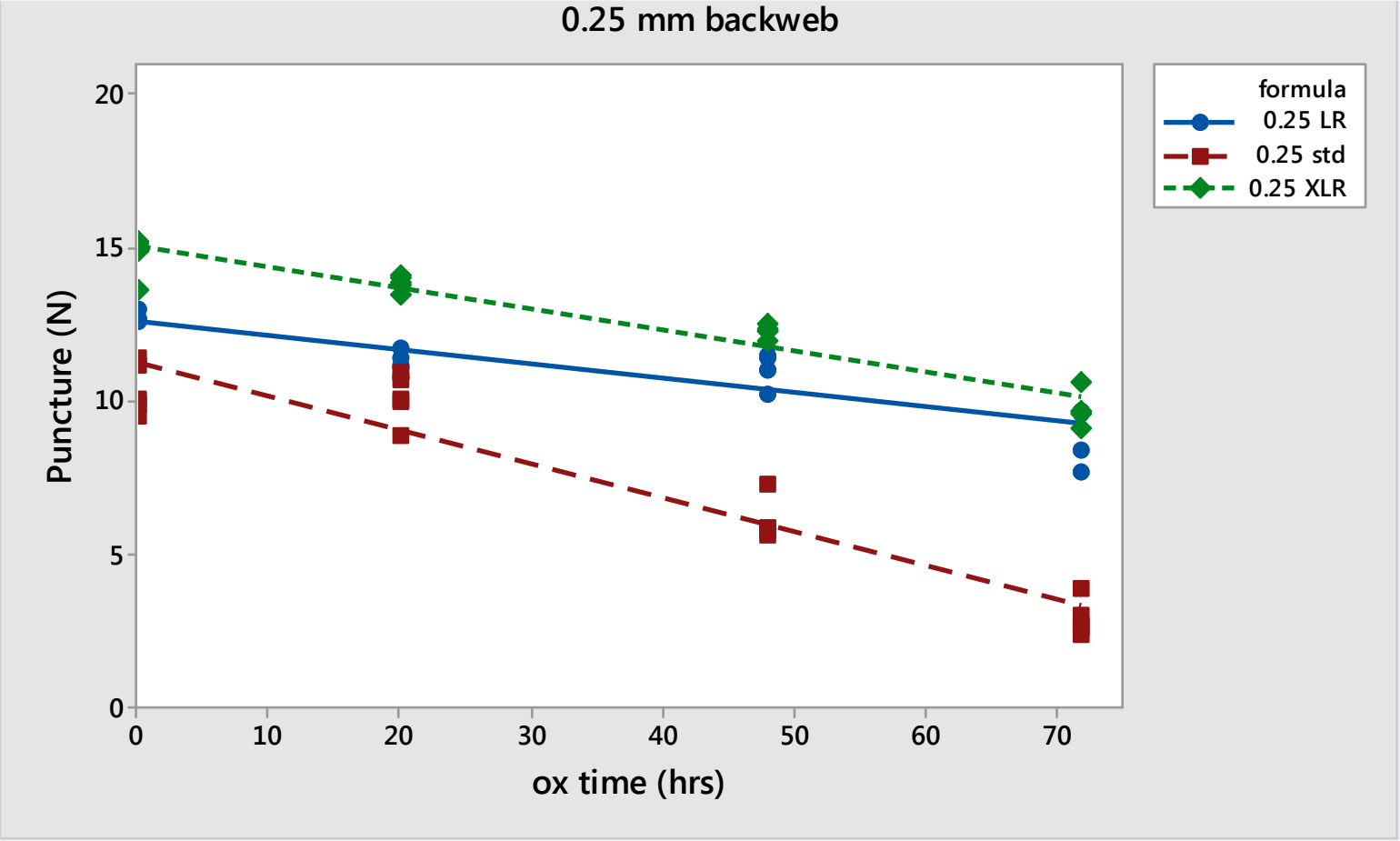
Polymer content : XLR > LR > STD

PEROX 80 --- % XMD ELONGATION VS EXPOSURE TIME



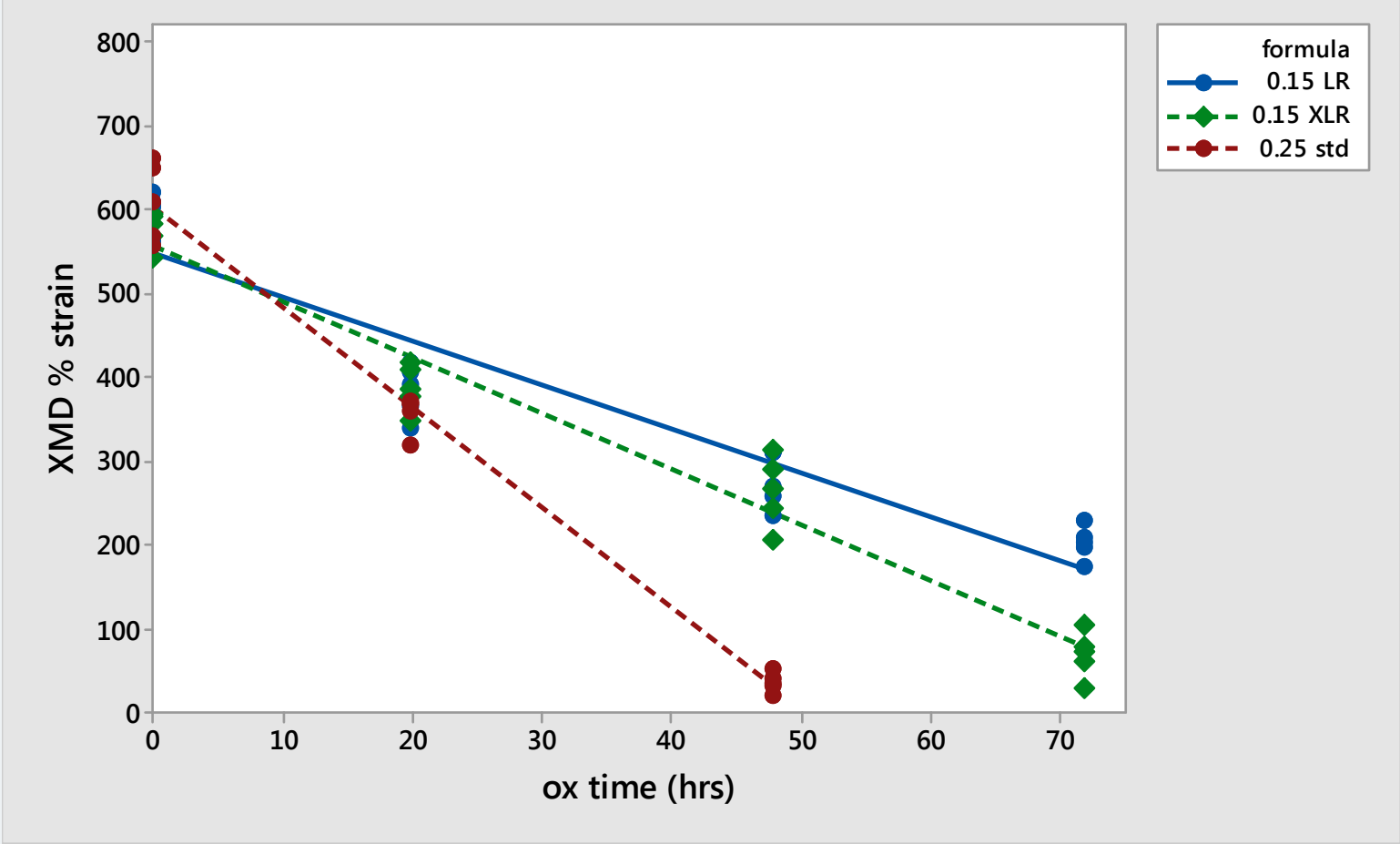
Polymer content : XLR > LR > STD

PEROX 80 --- PUNCTURE STRENGTH VS EXPOSURE TIME



Polymer content : XLR > LR > STD

CAN A 0.15 BW SEPARATOR BE DESIGNED TO HAVE AS GOOD OF OXIDATION RESISTANCE AS 0.25 BW?



Polymer content : XLR > LR > STD

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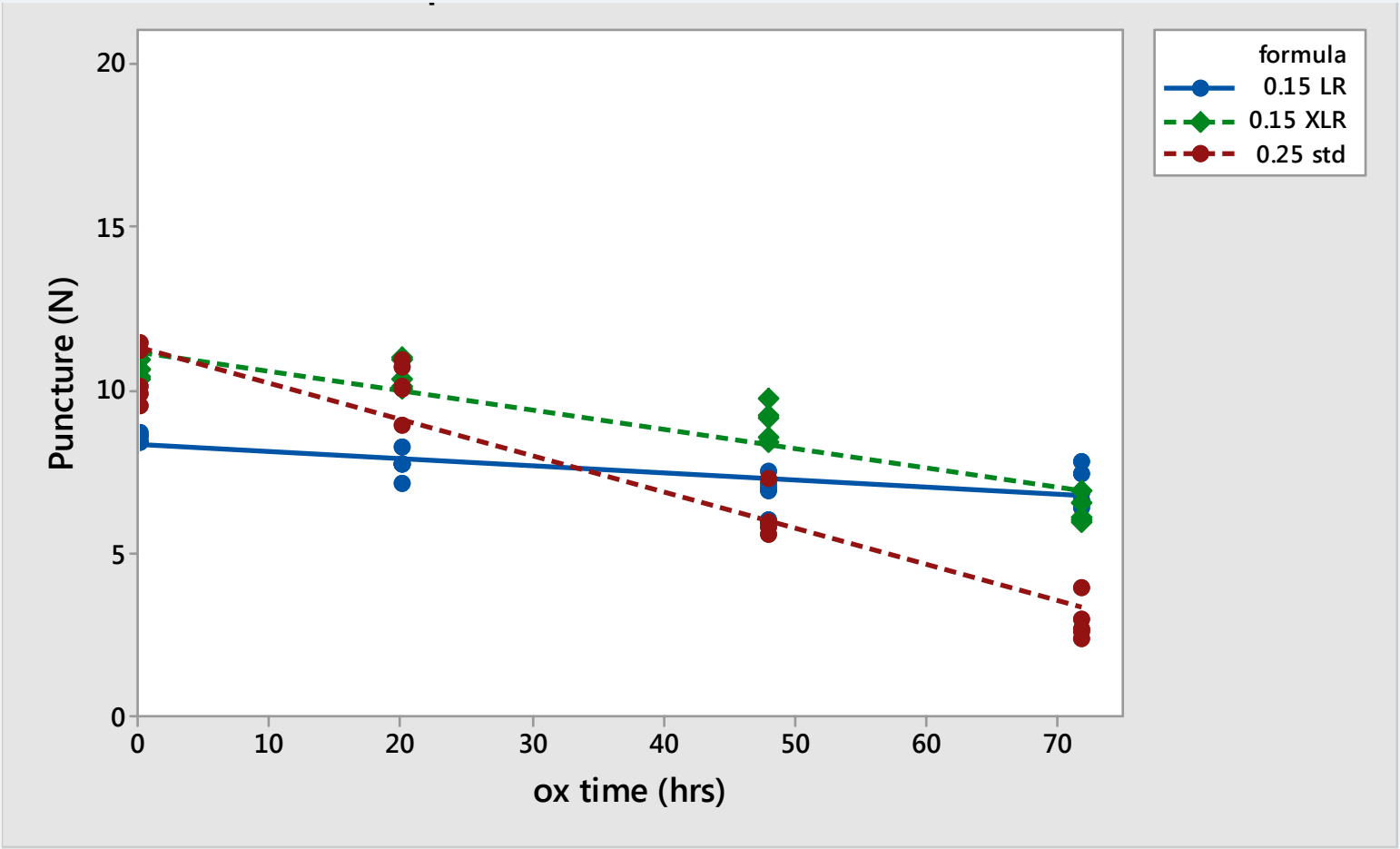
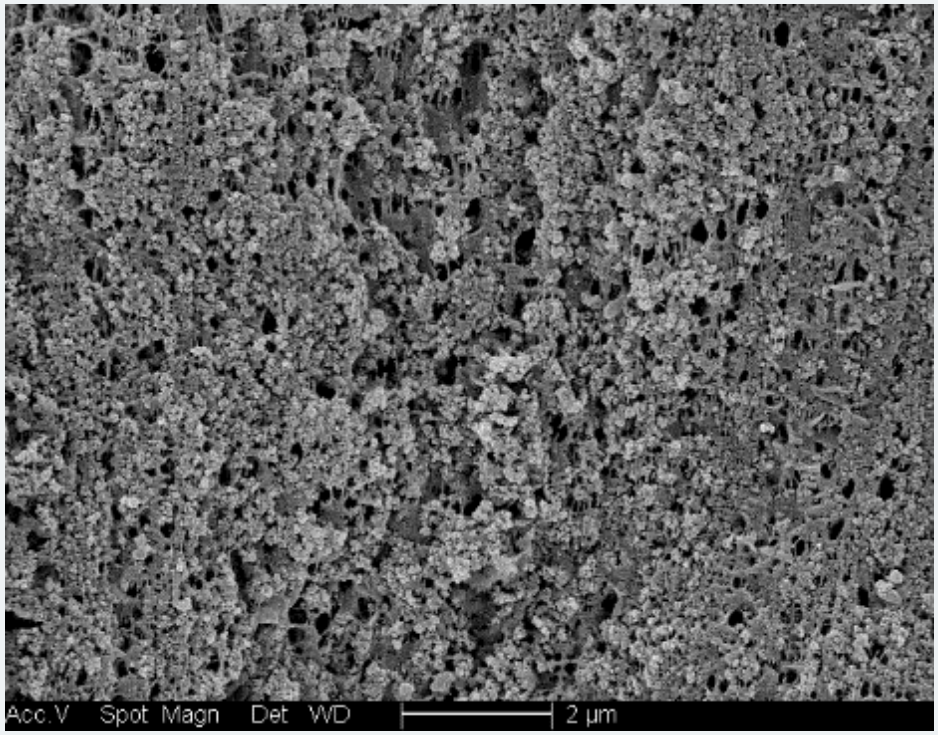
CAN A 0.15 BW SEPARATOR BE DESIGNED TO HAVE AS GOOD OF OXIDATION RESISTANCE AS 0.25 BW?



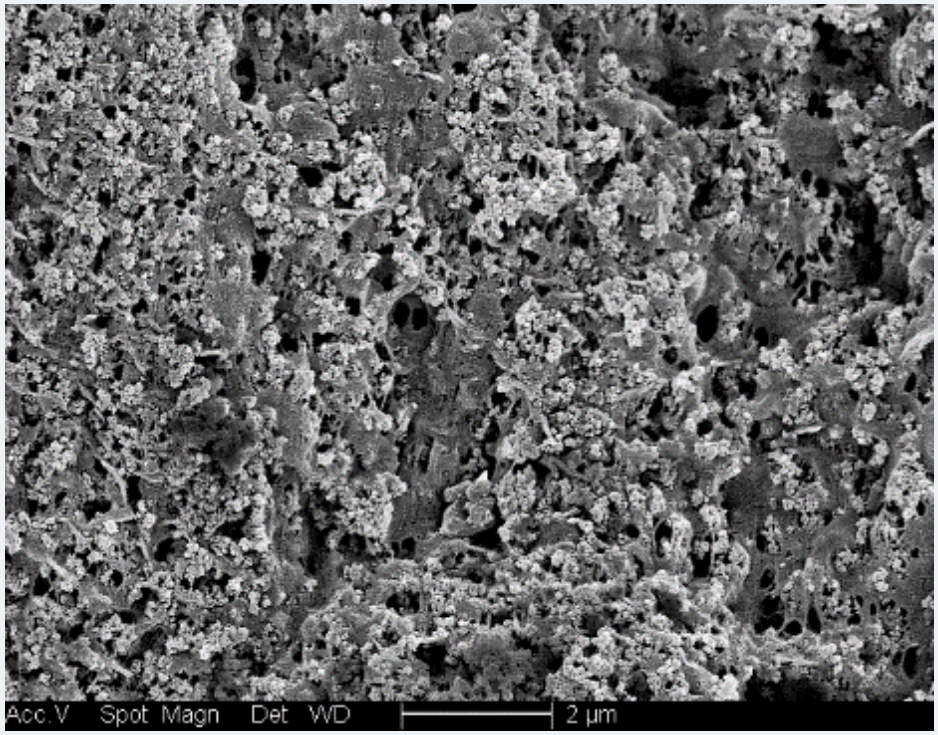
Polymer content : XLR > LR > STD

SURFACE SEM --- BEFORE AND AFTER PEROX 80 TEST

STD Separator



BEFORE

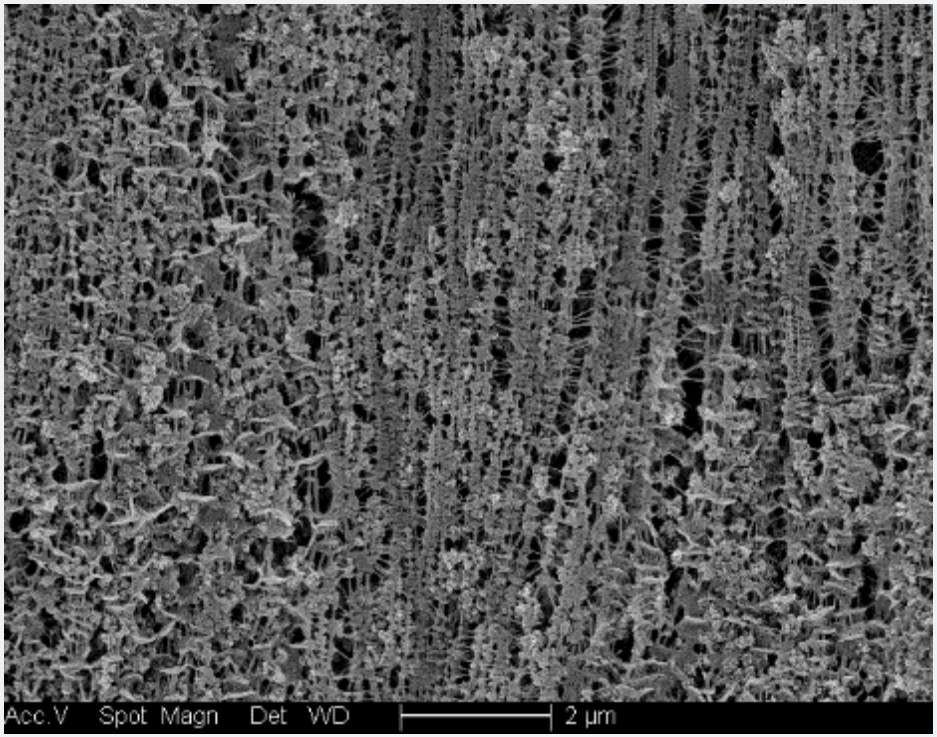


AFTER

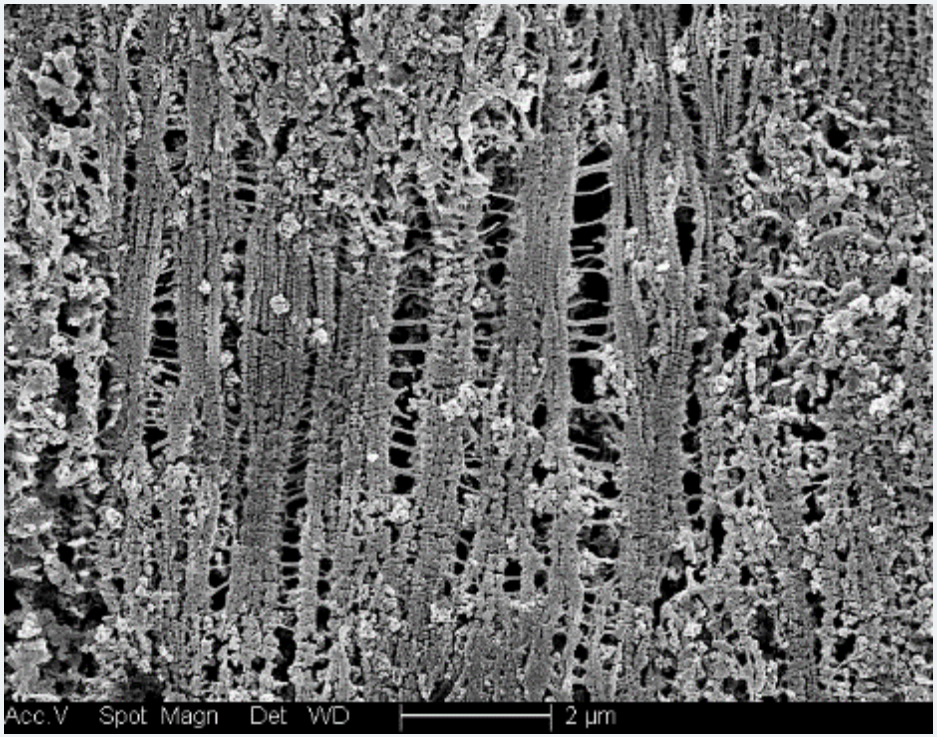
ENTEK

SURFACE SEM --- BEFORE AND AFTER PEROX 80 TEST

XLR Separator



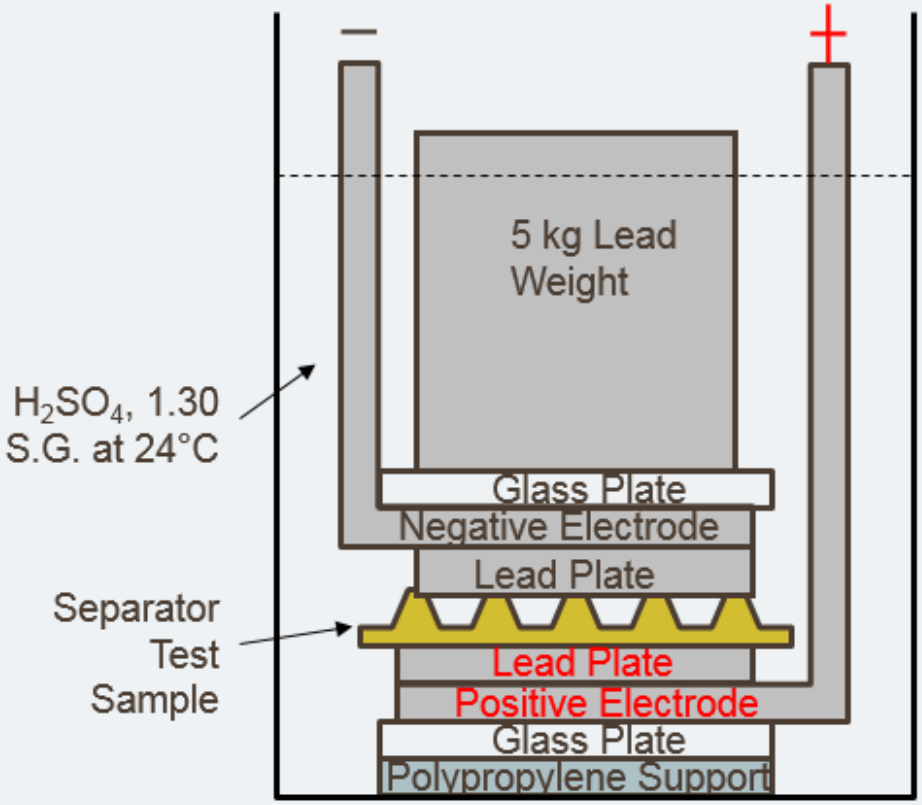
BEFORE

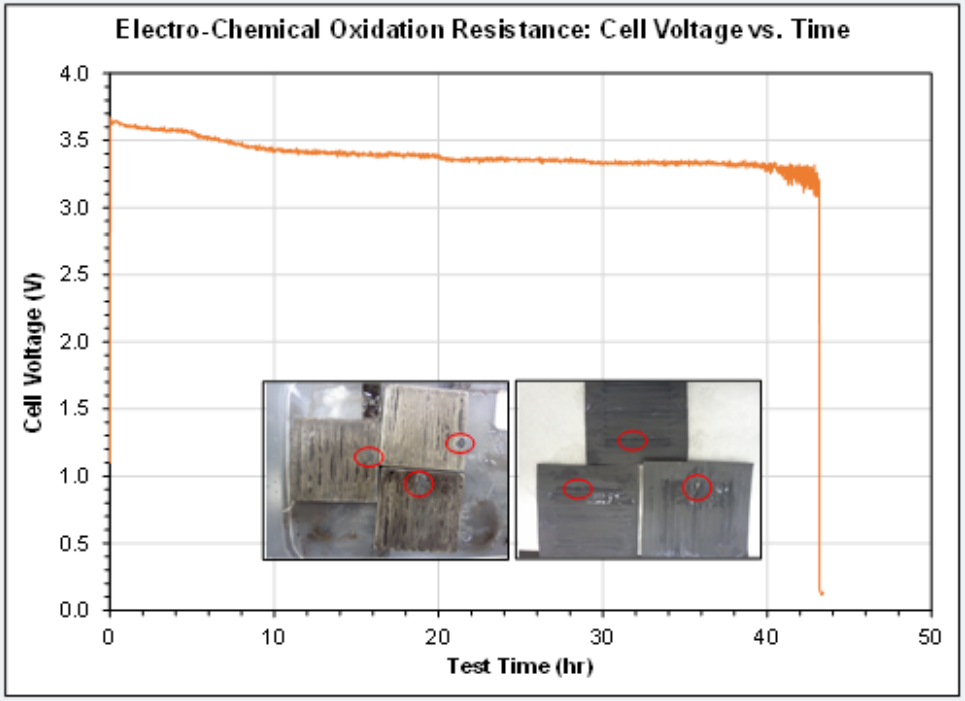


AFTER

ENTEK

ELECTROCHEMICAL OXIDATION TEST





- 5cm x 5cm specimens were overcharged with 200mA/cm² current between 2 pure lead plates at 75°C:
  - Three specimens per separator type
  - Samples "fail" when $\Delta V/\Delta t \geq 0.2$ V/min
- The polymer matrix within the separator is oxidized, allowing $PbSO_4$ to eventually grow to the positive plate, resulting in short-circuit.

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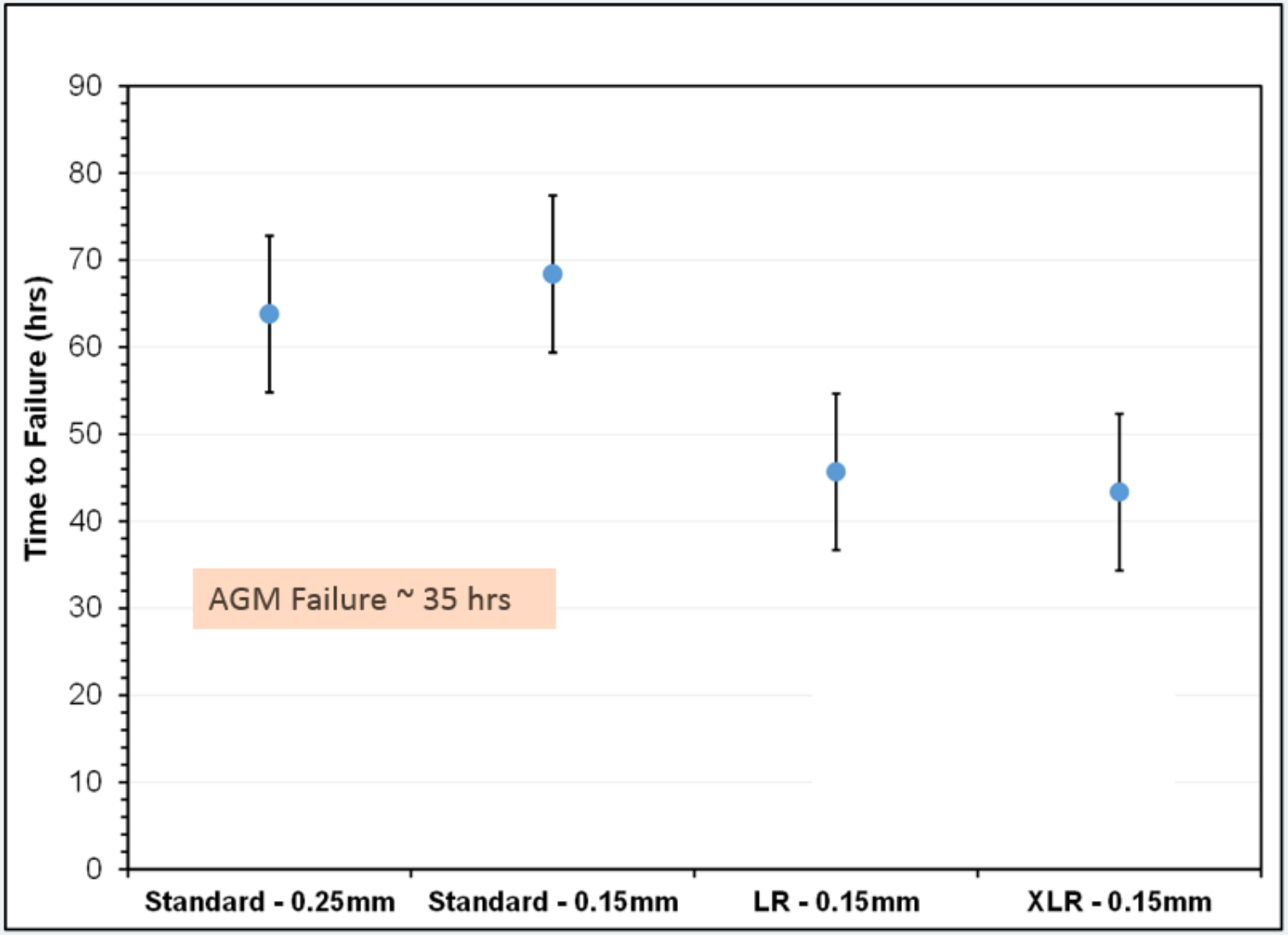
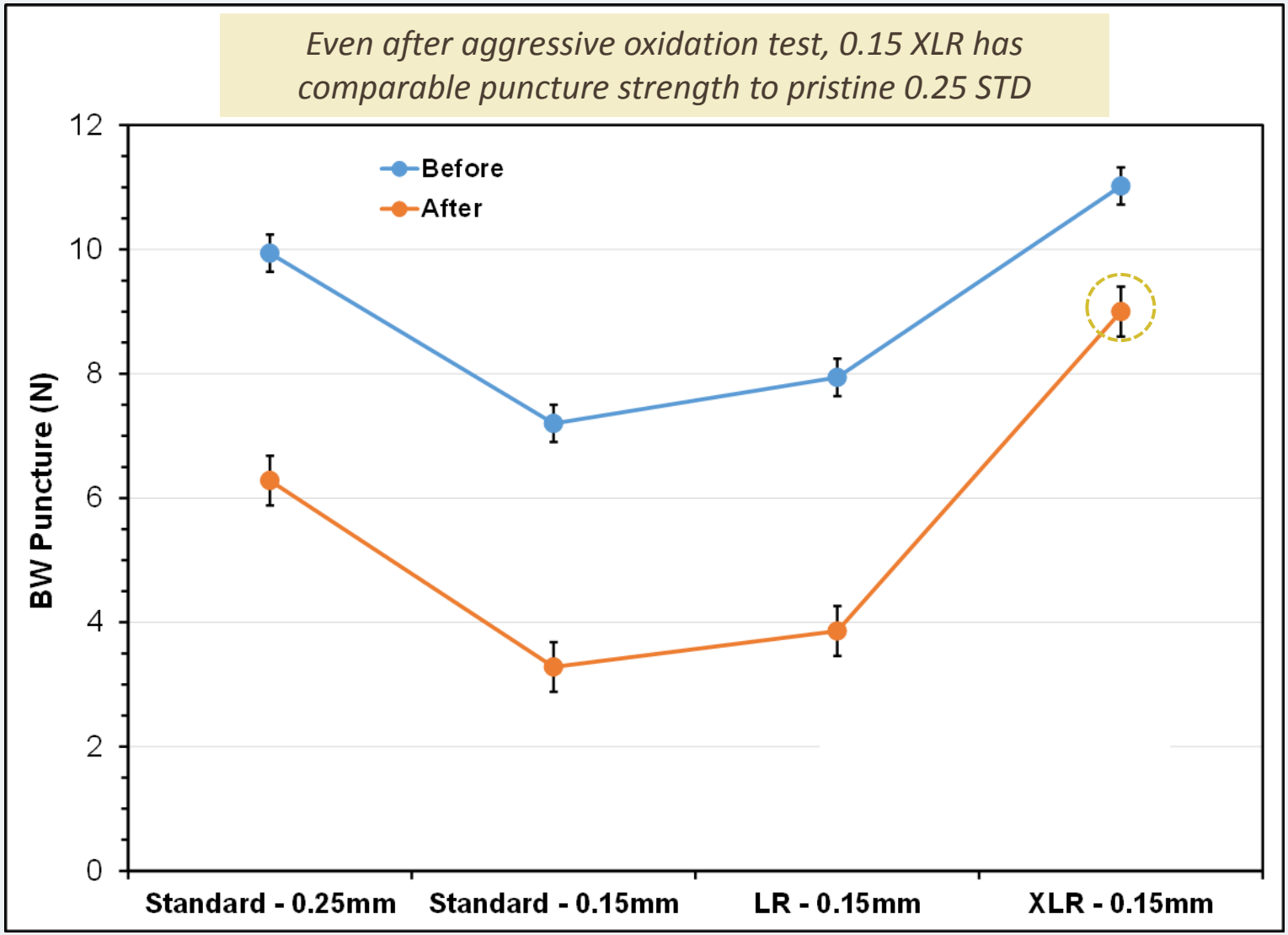
ELECTROCHEMICAL OXIDATION RESISTANCE



Polymer content : XLR > LR > STD

ELECTROCHEMICAL OXIDATION RESISTANCE (CONT'D)

Even after aggressive oxidation test, 0.15 XLR has comparable puncture strength to pristine 0.25 STD



Polymer content : XLR > LR > STD

SUMMARY

- PE/SiO_2 separators are susceptible to many chemical and physical changes both during their manufacture and within a Pb-acid battery environment
- The ***polymer content*** and ***oil/PE ratio*** are two critical parameters in the design and engineering of battery separators
- ENTEK has introduced a series of **LR** and **XLR** separators that have low electrical resistance, good mechanical properties, and excellent oxidation resistance
- Although some OEMs are reluctant to use thinner backweb separators in their battery designs, 0.15 XLR outperforms many 0.25 STD separators.

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