Characterization of the Electrical Properties of Contaminated Dielectric Oils for Pulsed-Power Research

S. L. RAMESH* and K. L. MARSHALL
University of Rochester, Laboratory for Laser Energetics
*Pittsford-Mendon High School and Summer High School Research Program

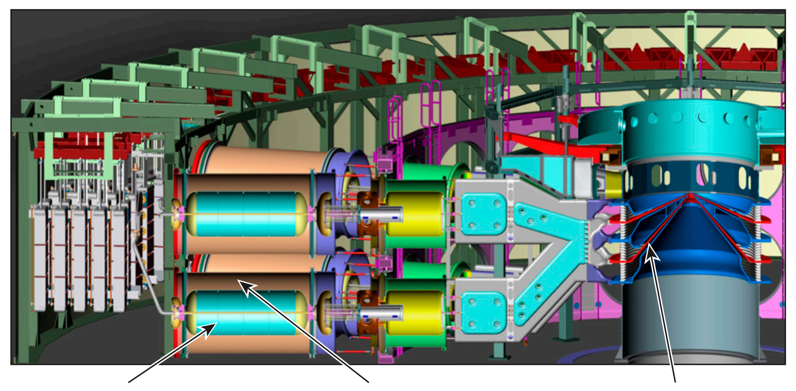
Summary

A purification process was developed for insulating oils used in pulsed-power research

- The effects of dielectric breakdown chemistry products on the electrical properties of Shell Diala S2-ZX-A insulating oil were assessed
  - the dielectric constant increases with contaminant level concentration
- "Molecular sieve" absorbant was found to be very effective in restoring the contaminated oil's resistivity
  - the purification process was evaluated using dc resistivity measurements and optical spectroscopy

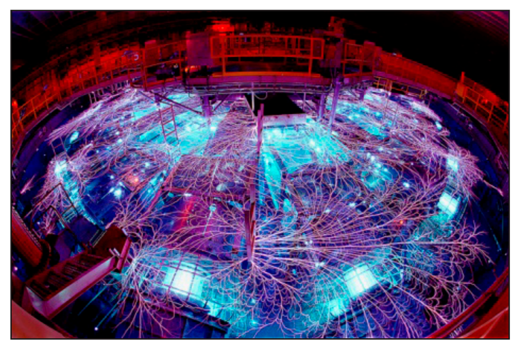
G11244

The electrical insulating oil in the Z-Machine becomes contaminated over time



- The purification process is scalable to varying volumes
  - Z-Machine (540,000 gal)
  - LLE's Pulsed-Power Laboratory (<1000 gal)
- The process is *in-situ* and continuous

Contamination comes from various sources



- Intrinsic:
  - dielectric breakdown
- Extrinsic:
  - water
  - dust particles
  - metal "grinds"

An absorption-based process employing "molecular sieve" was developed to purify the contaminated oil.

G11245

*http://www.sandia.gov/z-machine/about_z/how-z-works.html

Abstract

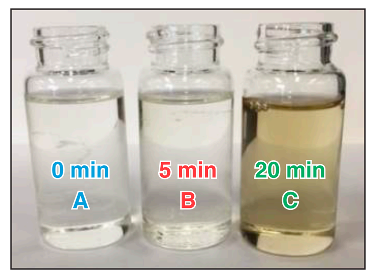
- The Z-machine at Sandia National Laboratories uses pulsed power to study magnetic confinement fusion. In the energy storage section, high-voltage capacitors are submerged in an insulating oil to minimize thermal and electric losses during system operation. Over time, this oil becomes contaminated with dielectric breakdown products resulting from high-voltage "flash-over" discharges and extrinsic contaminants (atmospheric moisture and dust particles) introduced into the oil during maintenance and cleaning.
- Impedance spectroscopy, dc resistivity/current flow measurements, and optical spectroscopy were used to evaluate the impact of contaminants on the electrical and optical properties of the insulating oil (Shell Diala S2 ZX-A). Samples contaminated with dielectric breakdown byproducts showed both a higher dielectric constant and a higher loss factor (tan δ) across a broad frequency range; the optical transmission also degraded as a function of contaminant concentration.
- Samples of Shell Diala S2 ZX-A insulating oil exposed to extended-interval, high-voltage electrical arc discharges were treated with activated carbon and type 13X molecular sieve absorbents in a laboratory-scale process designed to remove dielectric breakdown products. Both dc resistivity and optical transmission data were used to assess the efficiency of the purification process. Although both absorbents significantly improved the resistivity and optical transmission of the electrically degraded samples, the molecular sieve showed superior performance in restoring the dc resistivity. These results suggest that the contaminants are primarily charged species.
- A recirculating, cartridge-based absorption/filtration purification system employing molecular sieves was designed and proposed for continuous oil purification in the LLE Pulsed-Power Laboratory.

G11246

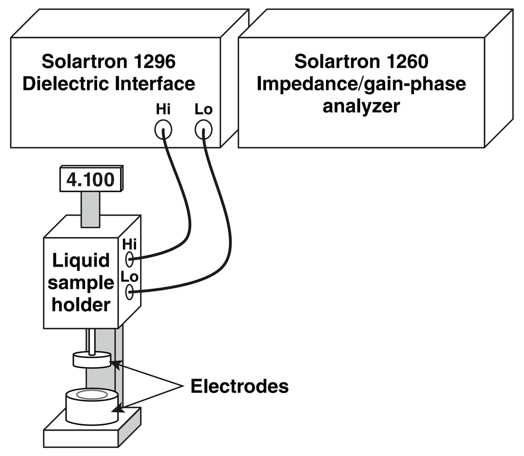
Dielectric Breakdown Analysis

An impedance/gain/phase analyzer was used to study the effects of dielectric breakdown on the insulating oil's frequency-response spectrum

High voltage (HV) discharge time

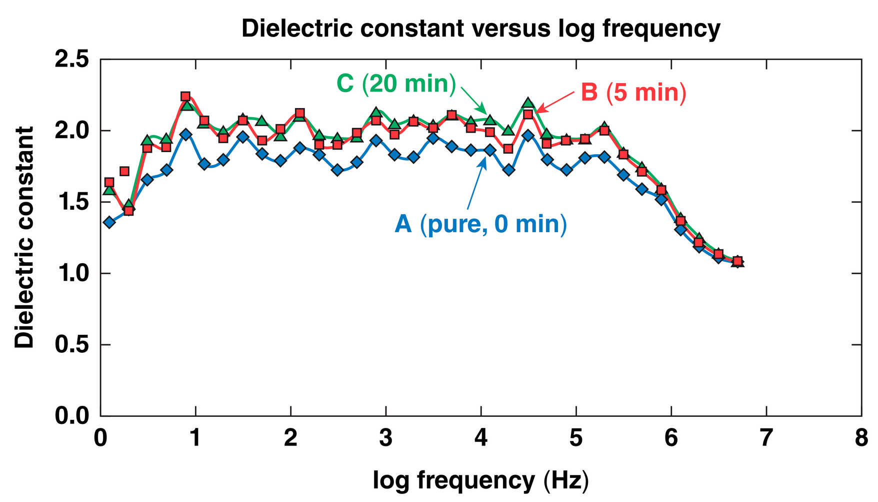


- 50-kV Tesla coil, 1-mm spark gap
- Sample volume: 5 mL



G11247

Dielectric breakdown products in the Shell Diala S2-ZX-A oil increases its dielectric constant over a broad frequency range



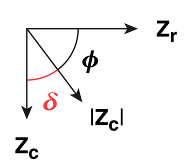
- Increased dielectric constant indicates a more-conductive nature
- No clear trend was found for lower frequencies (50 μHz to 1 Hz)

G11248

A similar trend was found in loss-factor measurements obtained from impedance spectroscopy

- The *loss factor* (tan δ) is calculated using the complement of the phase angle of the impedance

Arc discharge time (min)	Loss factor (tan δ) 50 Hz
0	0.0060
5	0.0173
20	0.0242



$$\text{Loss factor} = \frac{z_r}{z_c} = \tan\delta$$

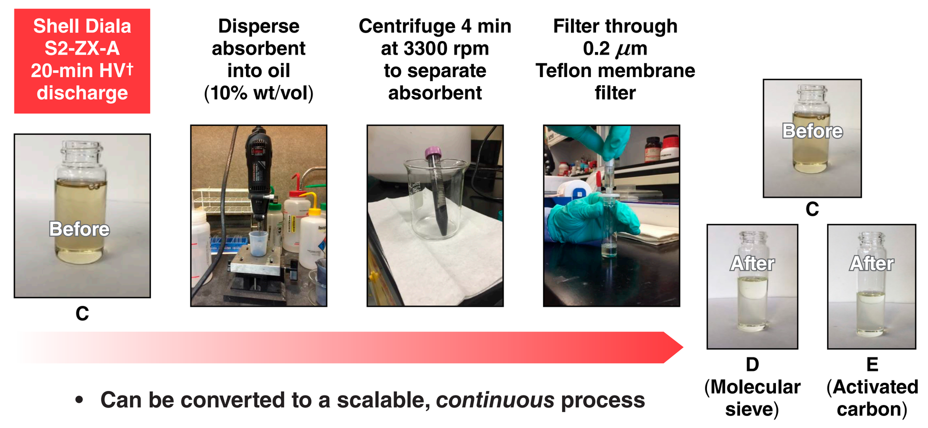
where δ = complementary angle to phase angle ϕ

- Impedance phase data was used to calculate and compare the tan δ values of the oil samples
- A large tan δ indicates greater energy loss under applied voltage and lower insulating capability

G11249

Purification Process Development

Both Type 13X molecular sieve* and activated carbon** were used as absorbents to purify the contaminated oil on a laboratory scale

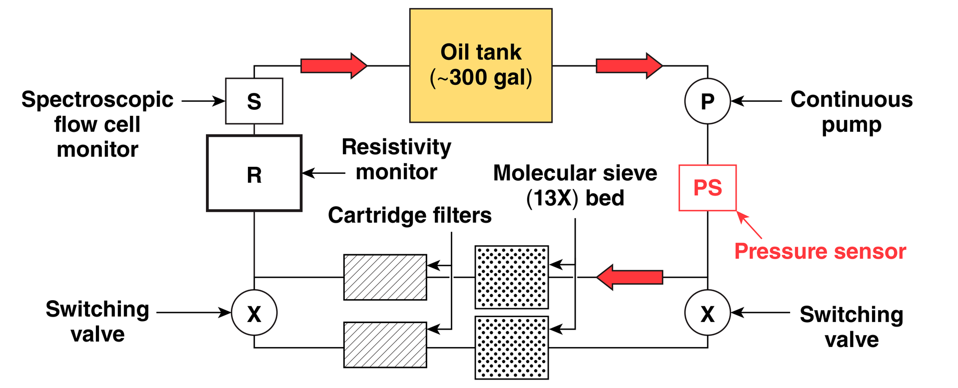


- Can be converted to a scalable, *continuous* process

G11250

*Chemical Dynamics Corporation (unactivated).
**University of Rochester, Department of Chemistry (Norit® activated carbon).
†HV: high voltage

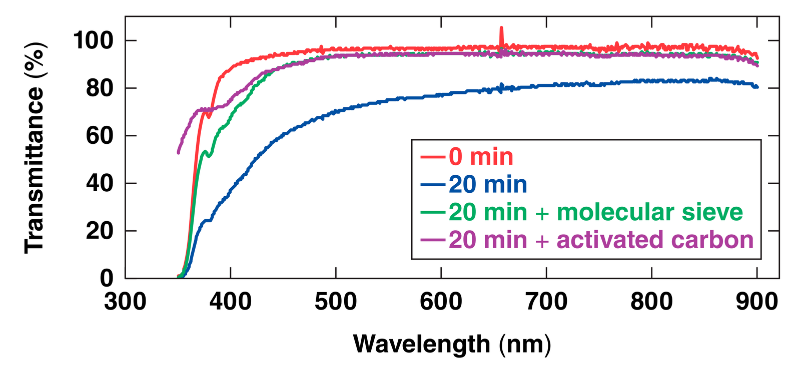
A recirculating cartridge-based absorption/filtration setup is proposed for the LLE Pulsed-Power Laboratory



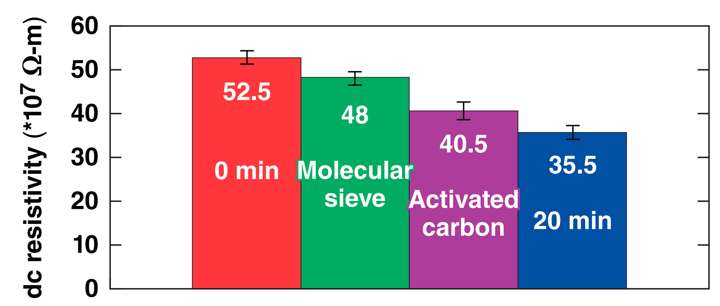
G11253

The dc resistivity* and transmittance spectra** were compared to evaluate the effectiveness of the purification process

Shell Diala S2-ZX-A, 20-min HV discharge



- Improved transmittance = less contamination



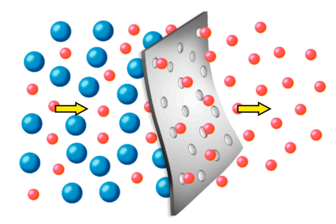
- Improved transmittance = greater insulating ability

G11251

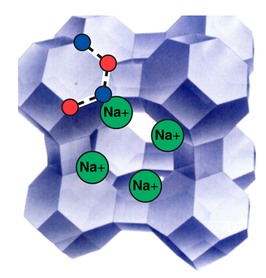
*Keithley 619 electrometer + YSI 3400 conductivity probe
**Agilent 8452 diode array spectrophotometer, 1 cm path length fused silica cell

Molecular sieves are more effective than activated carbon in removing dielectric breakdown contaminants

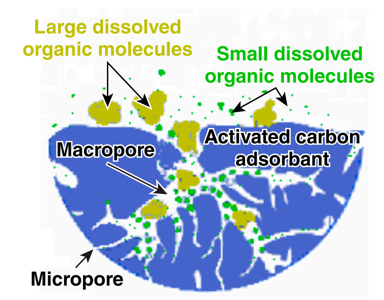
- Molecular sieves are highly porous aluminosilicate minerals with zeolite cage-like structures (Type 13X pore diameter = 10 Å)



- Zeolites have a highly charged internal field made of AlO_4 and SiO_4 tetrahedrons, which allow them to effectively absorb charged particles



- Activated carbon is an effective absorbent because of its high surface area (microporosity)
  - binds to materials using van der Waals intermolecular forces
- Does not work well with inorganic materials, metals, strong acids bases, and polar organics (e.g., alcohols)



G11252

http://srorzeolite.org/en/alsaeda-script/uploads/2014/08/Zeolite_Molecular_Sieve_4A-287x300.gif
http://medaad.com/wp-content/uploads/2016/02/a06_membrane_full.jpg
http://www.capitalcarbon.in/images/activated-carbon.gif

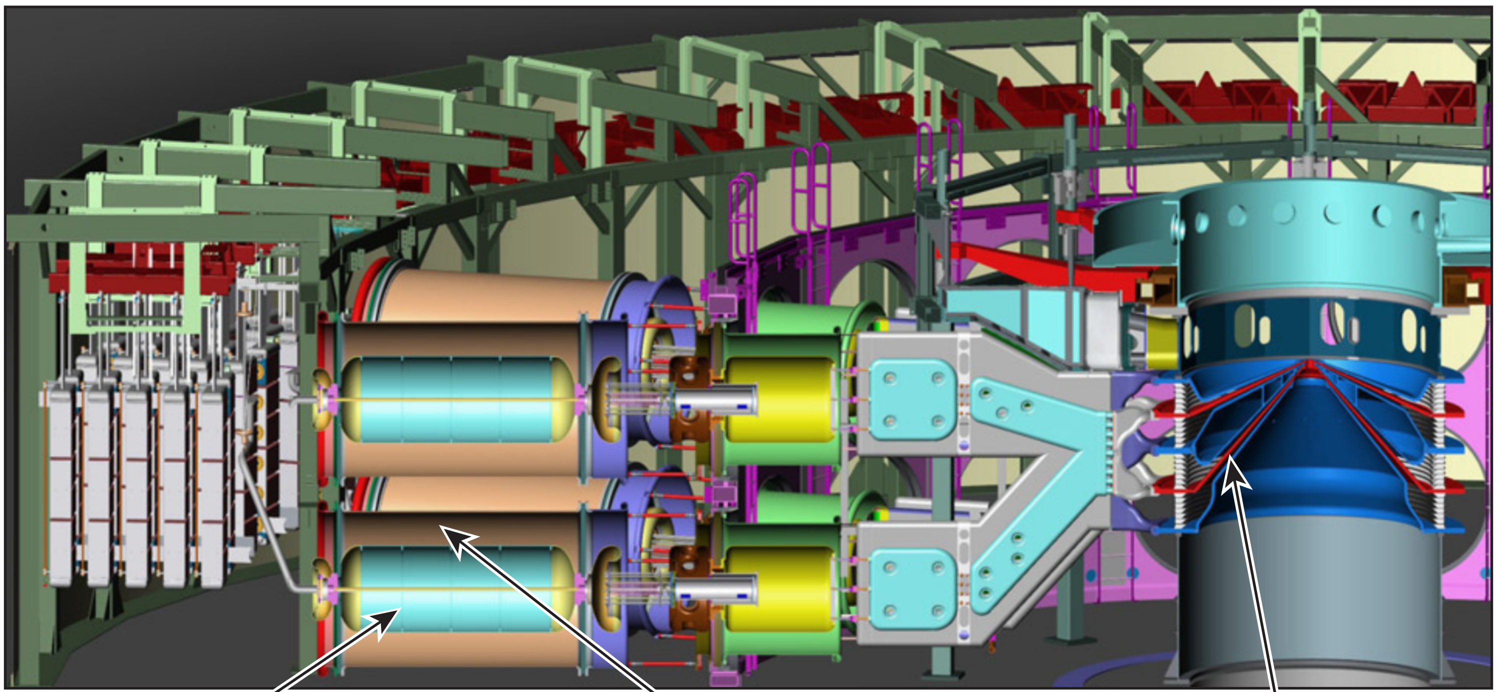
Summary

A purification process was developed for insulating oils used in pulsed-power research

- The effects of dielectric breakdown chemistry products on the electrical properties of Shell Diala S2-ZX-A insulating oil were assessed
  - the dielectric constant increases with contaminant level concentration
- "Molecular sieve" absorbant was found to be very effective in restoring the contaminated oil's resistivity
  - the purification process was evaluated using dc resistivity measurements and optical spectroscopy

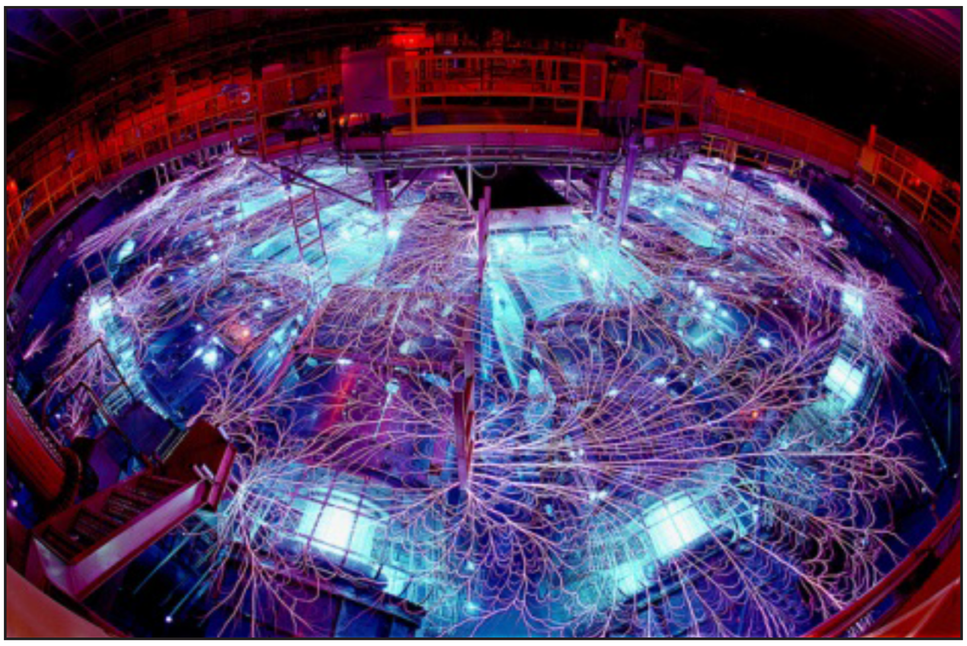
G11244

The electrical insulating oil in the Z-Machine becomes contaminated over time



- The purification process is scalable to varying volumes
  - Z-Machine (540,000 gal)
  - LLE's Pulsed-Power Laboratory (<1000 gal)
- The process is *in-situ* and continuous

Contamination comes from various sources



- Intrinsic:
  - dielectric breakdown
- Extrinsic:
  - water
  - dust particles
  - metal "grinds"

An absorption-based process employing "molecular sieve" was developed to purify the contaminated oil.

G11245

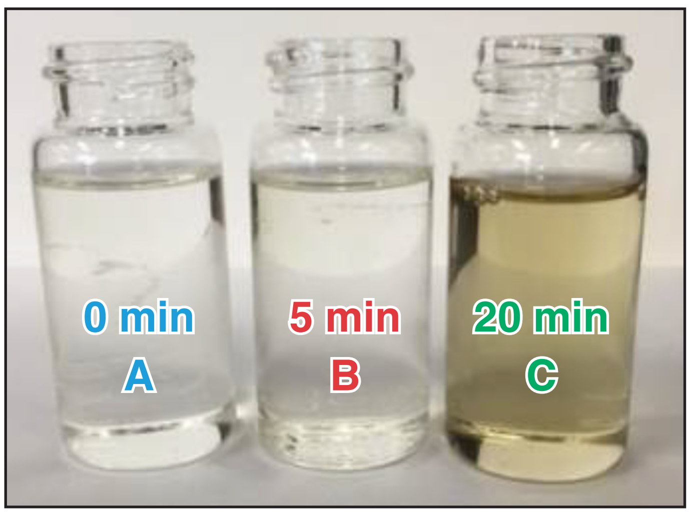
*http://www.sandia.gov/z-machine/about_z/how-z-works.html

Abstract

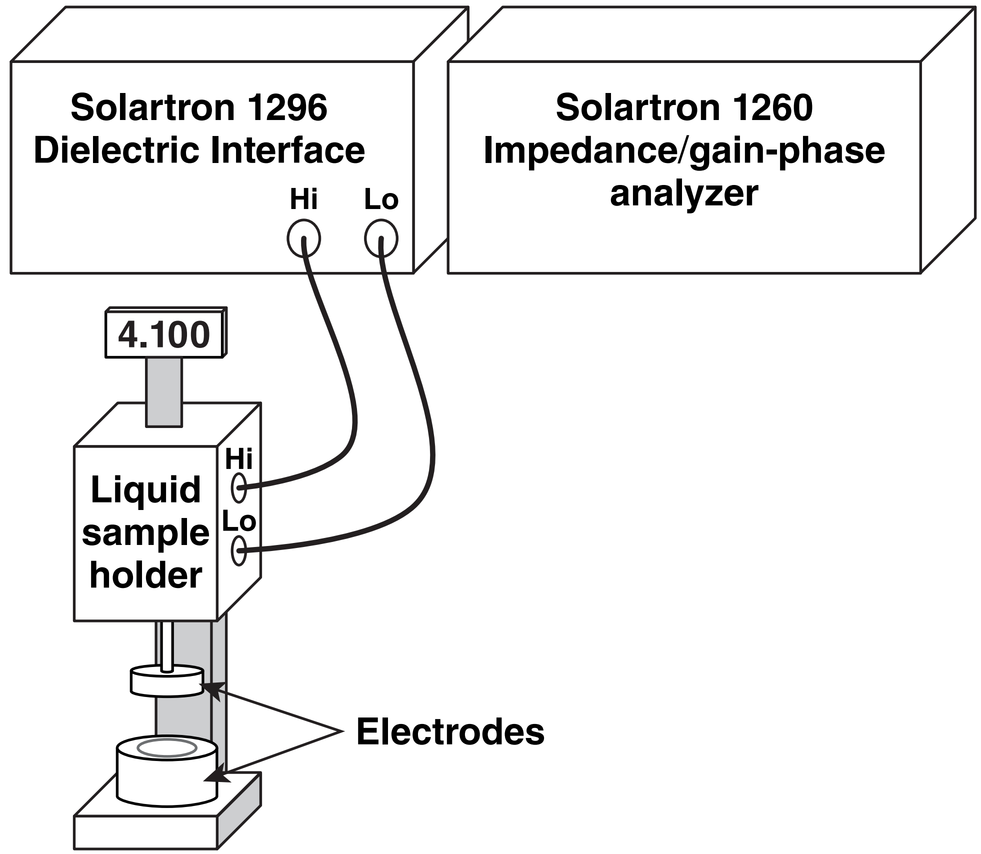
- The Z-machine at Sandia National Laboratories uses pulsed power to study magnetic confinement fusion. In the energy storage section, high-voltage capacitors are submerged in an insulating oil to minimize thermal and electric losses during system operation. Over time, this oil becomes contaminated with dielectric breakdown products resulting from high-voltage "flash-over" discharges and extrinsic contaminants (atmospheric moisture and dust particles) introduced into the oil during maintenance and cleaning.
- Impedance spectroscopy, dc resistivity/current flow measurements, and optical spectroscopy were used to evaluate the impact of contaminants on the electrical and optical properties of the insulating oil (Shell Diala S2 ZX-A). Samples contaminated with dielectric breakdown byproducts showed both a higher dielectric constant and a higher loss factor (tan δ) across a broad frequency range; the optical transmission also degraded as a function of contaminant concentration.
- Samples of Shell Diala S2 ZX-A insulating oil exposed to extended-interval, high-voltage electrical arc discharges were treated with activated carbon and type 13X molecular sieve absorbents in a laboratory-scale process designed to remove dielectric breakdown products. Both dc resistivity and optical transmission data were used to assess the efficiency of the purification process. Although both absorbents significantly improved the resistivity and optical transmission of the electrically degraded samples, the molecular sieve showed superior performance in restoring the dc resistivity. These results suggest that the contaminants are primarily charged species.
- A recirculating, cartridge-based absorption/filtration purification system employing molecular sieves was designed and proposed for continuous oil purification in the LLE Pulsed-Power Laboratory.

An impedance/gain/phase analyzer was used to study the effects of dielectric breakdown on the insulating oil's frequency-response spectrum

High voltage (HV) discharge time



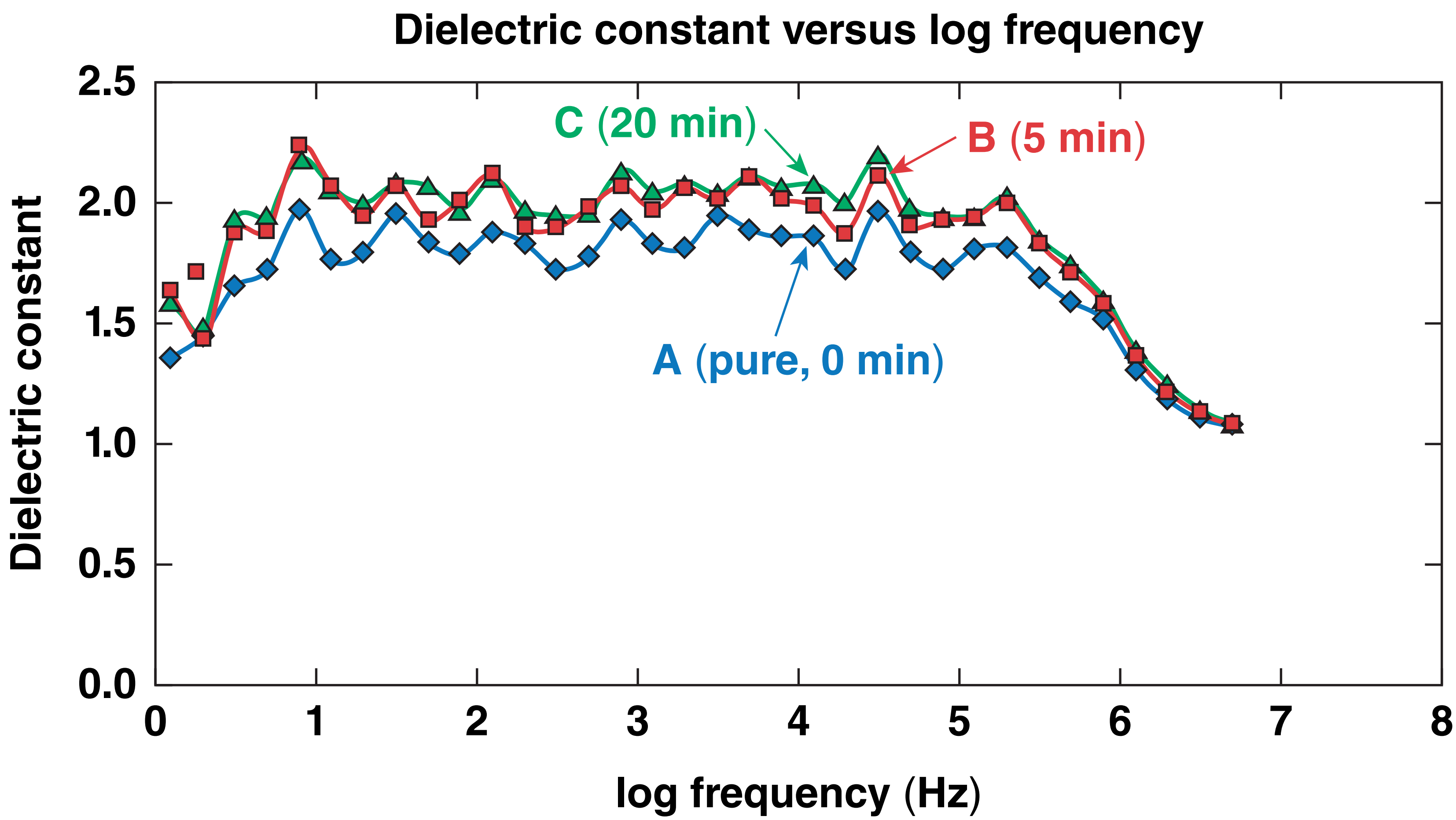
- 50-kV Tesla coil, 1-mm spark gap
- Sample volume: 5 mL



G11247

Dielectric breakdown products in the Shell Diala S2-ZX-A oil increases its dielectric constant over a broad frequency range

UR LLE



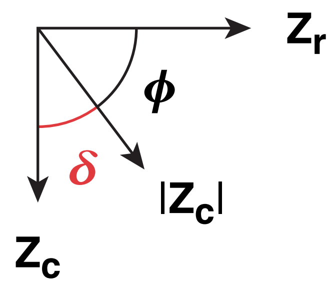
- Increased dielectric constant indicates a more-conductive nature
- No clear trend was found for lower frequencies (50 μHz to 1 Hz)

G11248

A similar trend was found in loss-factor measurements obtained from impedance spectroscopy

- The *loss factor* (tan δ) is calculated using the complement of the phase angle of the impedance

Arc discharge time (min)	Loss factor (tan δ) 50 Hz
0	0.0060
5	0.0173
20	0.0242



$$\text{Loss factor} = \frac{Z_r}{Z_c} = \tan\delta$$

where δ = complementary angle to phase angle ϕ

- Impedance phase data was used to calculate and compare the tan δ values of the oil samples
- A large tan δ indicates greater energy loss under applied voltage and lower insulating capability

G11249

Both Type 13X molecular sieve* and activated carbon** were used as absorbents to purify the contaminated oil on a laboratory scale

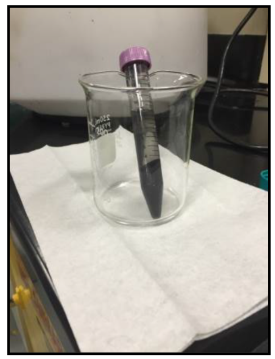
UR LLE

Shell Diala S2-ZX-A 20-min HV† discharge

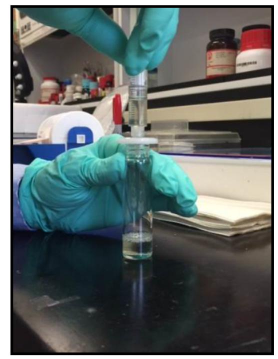
C

Disperse absorbent into oil (10% wt/vol)

Centrifuge 4 min at 3300 rpm to separate absorbent

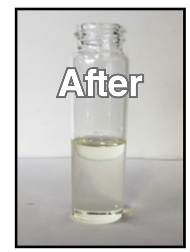


Filter through 0.2 μm Teflon membrane filter



C

D (Molecular sieve)



E (Activated carbon)

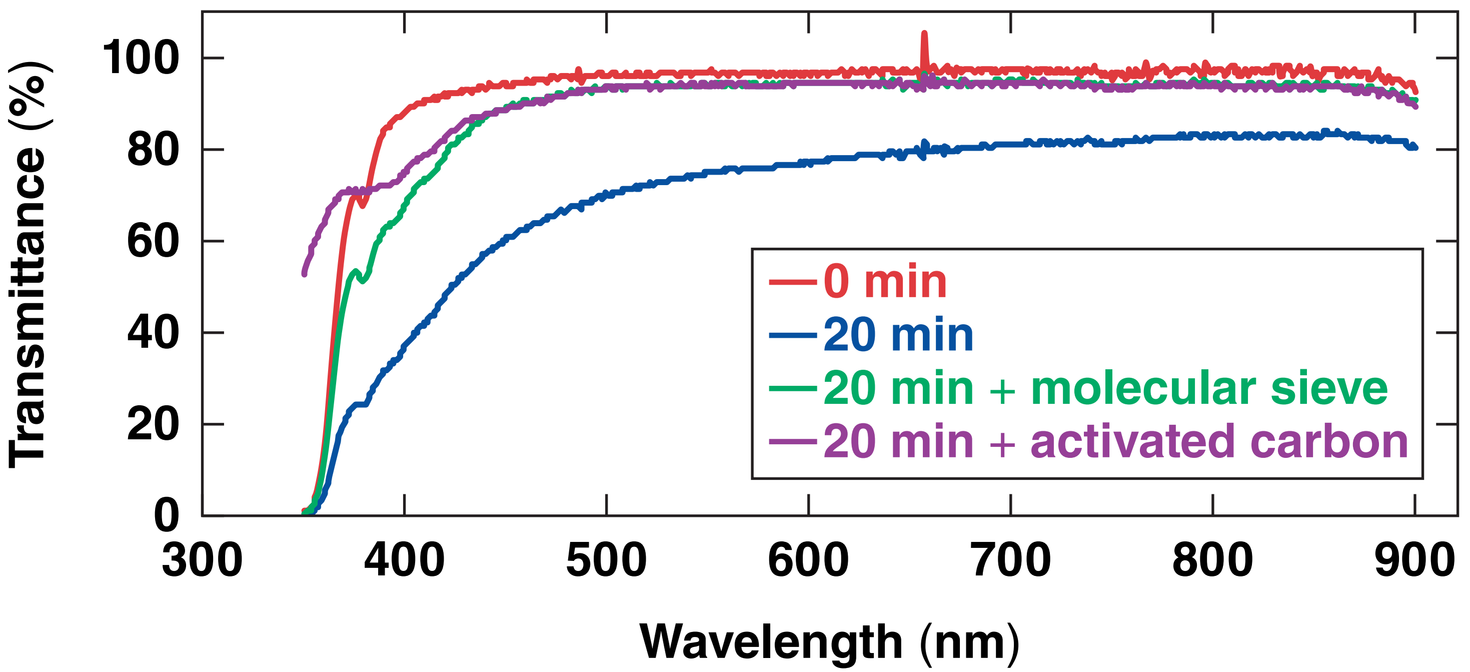
- Can be converted to a scalable, *continuous* process

*Chemical Dynamics Corporation (unactivated).
**University of Rochester, Department of Chemistry (Norit® activated carbon).
†HV: high voltage

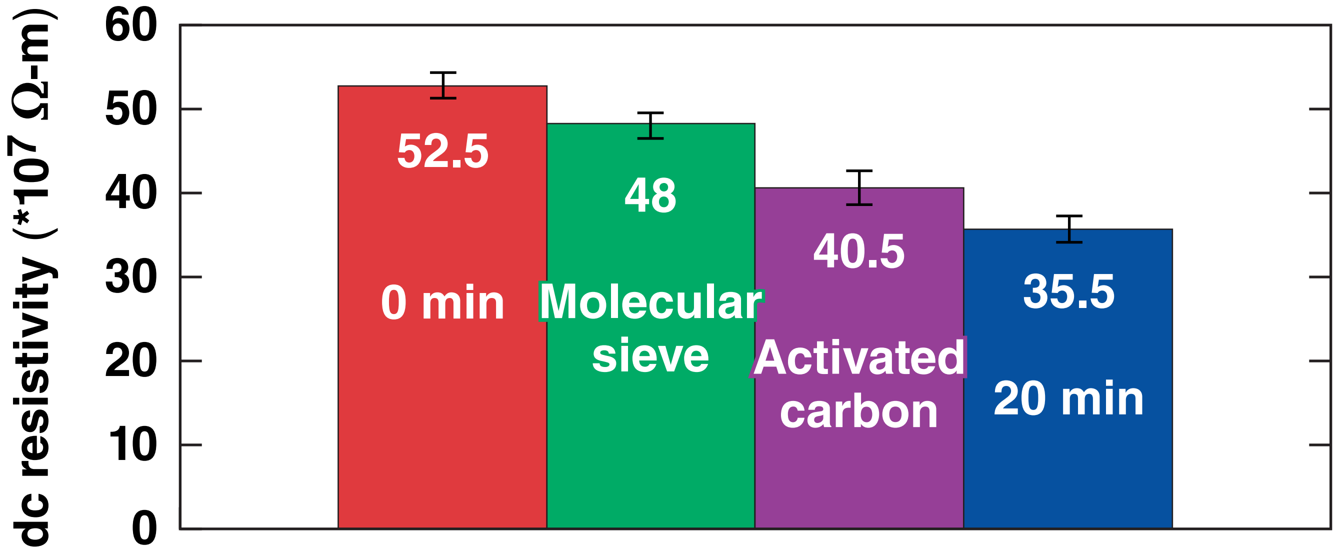
G11250

The dc resistivity* and transmittance spectra** were compared to evaluate the effectiveness of the purification process

Shell Diala S2-ZX-A, 20-min HV discharge



- Improved transmittance = less contamination



- Improved transmittance = greater insulating ability

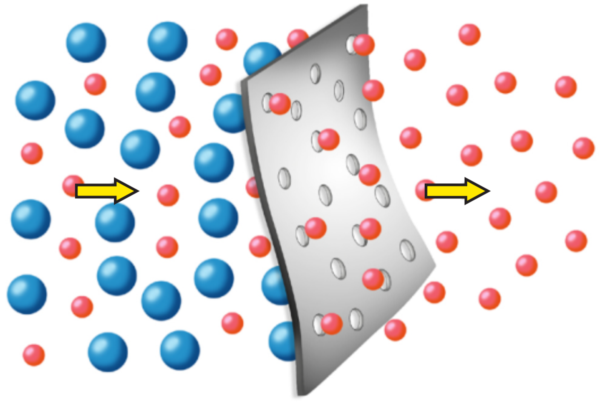
G11251

*Keithley 619 electrometer + YSI 3400 conductivity probe

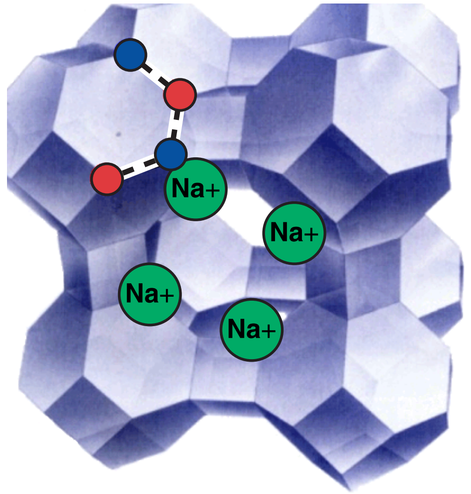
**Agilent 8452 diode array spectrophotometer, 1 cm path length fused silica cell

Molecular sieves are more effective than activated carbon in removing dielectric breakdown contaminants

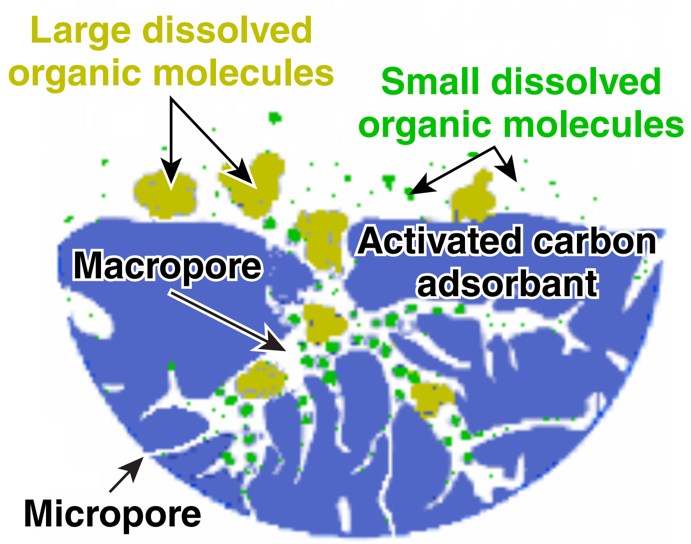
- Molecular sieves are highly porous aluminosilicate minerals with zeolite cage-like structures (Type 13X pore diameter = 10 Å)



- Zeolites have a highly charged internal field made of AlO_4 and SiO_4 tetrahedrons, which allow them to effectively absorb charged particles

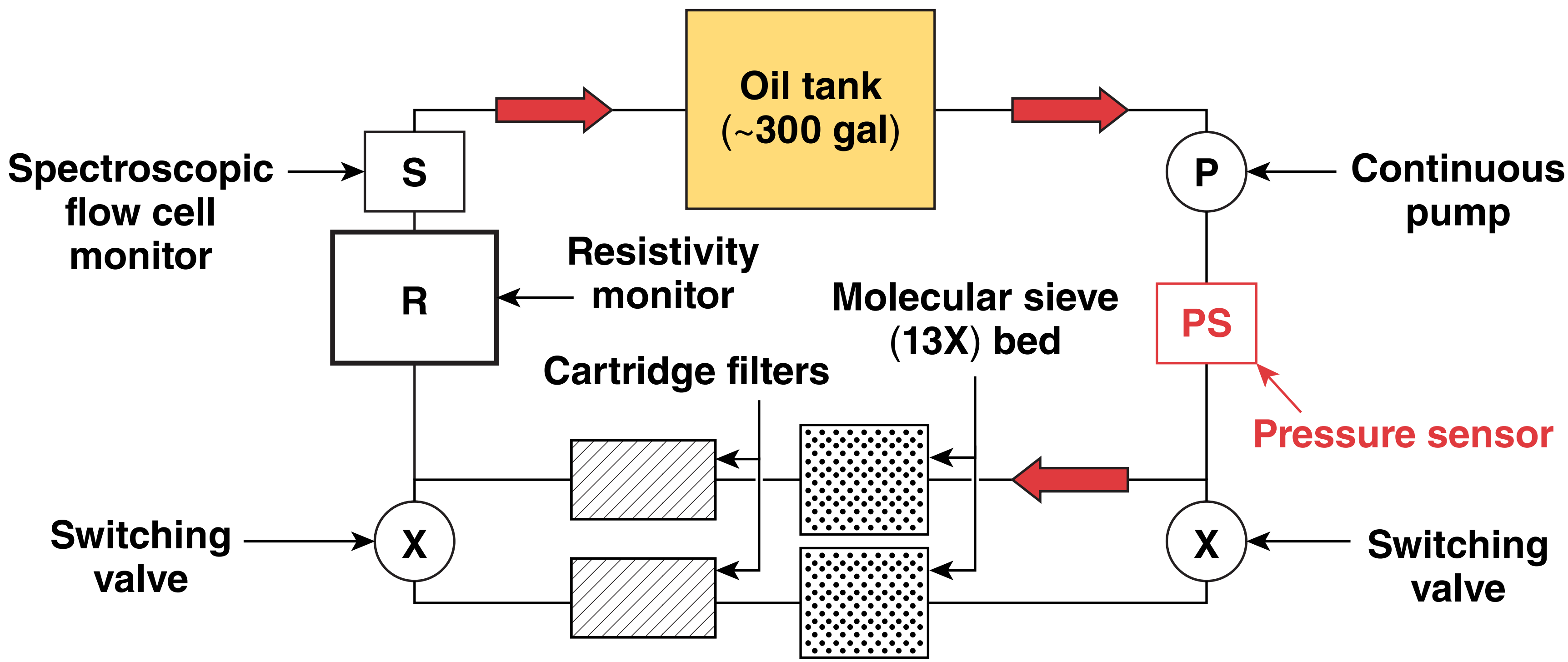


- Activated carbon is an effective absorbent because of its high surface area (microporosity)
  - binds to materials using van der Waals intermolecular forces
- Does not work well with inorganic materials, metals, strong acids bases, and polar organics (e.g., alcohols)



http://srorzeolite.org/en/alsaeda-script/uploads/2014/08/Zeolite_Molecular_Sieve_4A-287x300.gif
http://medaad.com/wp-content/uploads/2016/02/a06_membrane_full.jpg
http://www.capitalcarbon.in/images/activated-carbon.gif

A recirculating cartridge-based absorption/filtration setup is proposed for the LLE Pulsed-Power Laboratory



G11253