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Fabrication of Single-walled Carbon Nanotube (SWNT) Sorbent for Volatile Organic Compounds (VOCs) Sampling

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INTRODUCTION:
VOCs Sampling and Analysis

SKC Anasorb CSC Sorbent Tube

- Sampling (Adsorption)
  - (Ad)sorbent: Charcoal or Activated Carbon
    - High adsorption capacity & affordable
- Analysis (Desorption and Measurement)
  - Chemical extraction
    - Long desorption time (i.e., > 30 min) in a toxic solvent
    - Somewhat expensive GC (gas chromatography) vials
    - Low sensitivity
  - Thermal extraction
    - Desorption directly in GC system; much more sensitive
    - Reliability issue (e.g., poor sealing)
    - Expensive system

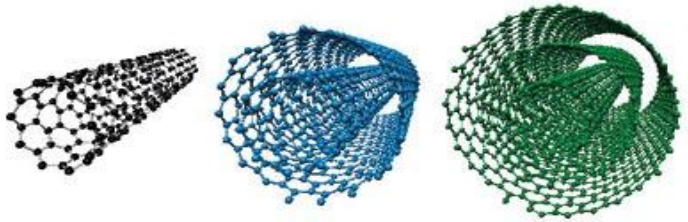
INTRODUCTION:
New Desorption Technique

- PhotoThermal Desorption (PTD)
  - Visible light (photo flash) thermally desorbing an analyte & quantified by photoionization detector
  - An alternative for VOCs analysis of *passive sampler*
  - Elimination of sample preparation time and cost
  - Bridging the sensitivity gap between chemical desorption and thermal desorption

INTRODUCTION: Potential Sorbent

Carbon Nanotubes (CNTs)

- *Large surface area* (300-1,000 m^2/g)
  - Tunable pore diameter by fabrication process



One, Two, and Three walls of CNTs

- *Superior thermal*, electrical, optical conductivity
  - A wide variety of applications (e.g., mechanical, structural, thermal, electrical, biomedical, etc.)

- Single-walled Carbon Nanotubes (SWNTs)
  - Considered as prime material for gas sorption
  - Higher in thermal/electrical conductivity (3000 W/m·K)[1]

[1] Thermal conductivity of gold: 310 W/m·K

PURPOSE

- Develop a CNT sorbent coupled with photothermal desorption technique to be used in a VOC passive sampler
  - Investigate fabrication methods of SWNTs to a self-standing form (i.e., buckypaper)
  - Examine adsorption efficiencies (i.e., surface area, pore size, and toluene adsorption isotherm)

METHODS:
Fabrication of Buckypaper (BP)

- Arc Discharge (AD) SWNT solution[1]

- Suspension and filtration
  - Suspension with solvents/surfactants (i.e., acetone and methanol)
  - Vacuum filtration
  - Additional cleaning with deionized water and solvents[2]

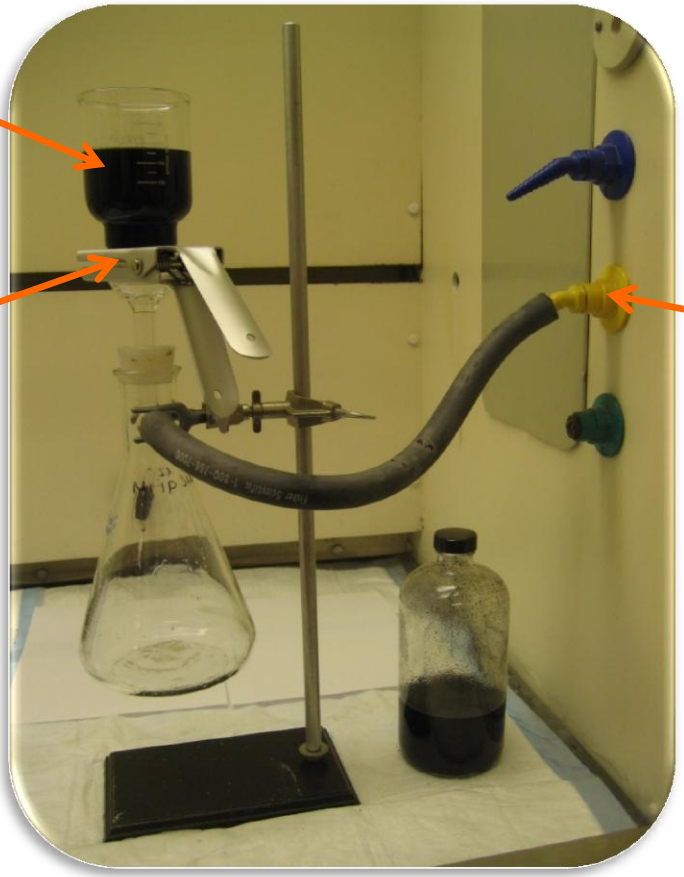
- Three types of BP samples (50 mg each, n=2)
  - Non-cleaned (acetone suspension[3] without cleaning)
  - Acetone-cleaned (acetone suspension[3] with cleaning)
  - Methanol-cleaned (methanol suspension[3] with cleaning)

[1] Suspended in 1 % surfactants (i.e., sodium cholate and sodium dodecyl sulfate)
[2] Repeated 2 times with 250 mL deionized water and 50 mL solvent
[3] 15 hrs of suspension in 400 mL solvent

METHODS

SWNTs suspended in solvent

Membrane filter



Vacuum

METHODS: Adsorption Efficiency

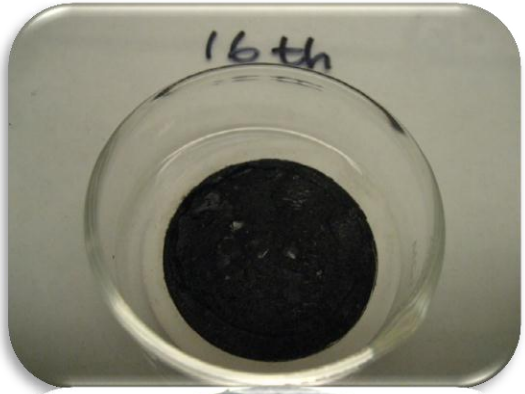
- Surface area & Pore size
  - ASAP2020 Physisorption Analyzer
  - Nitrogen as adsorbate
  - 77 K (-196 ˚C)

- Toluene Adsorption Isotherm[1]
  - Physisorption Analyzer
  - Toluene as adsorbate
  - 25 ˚C

[1] Analyzed at Micromeritics® because of a special set-up required

RESULTS:
Fabrication of Buckypaper

- Non-cleaned BP
- Acetone-cleaned BP
- Methanol-cleaned BP



RESULTS: Adsorption Efficiency

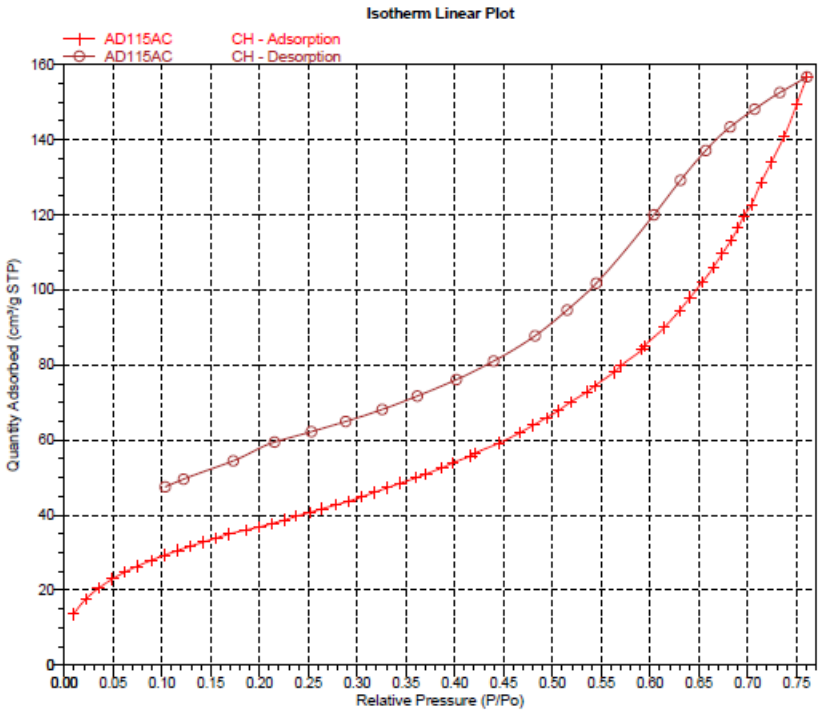
- Surface area[1] and Pore size of Arc Discharge[2] BP

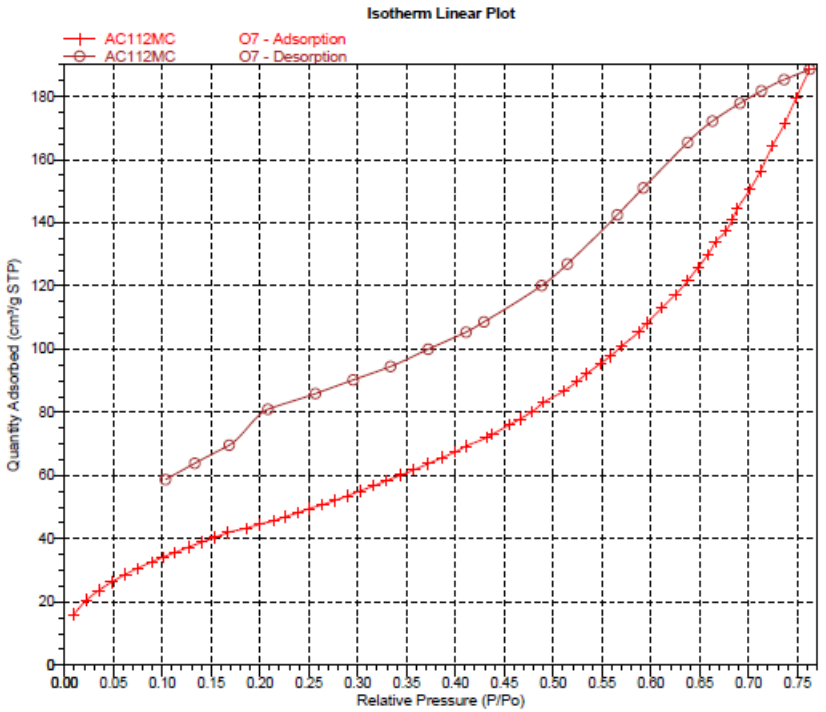
Fabrication Method	NOT-CLEANED BP (Acetone suspension & Filtration w/o Cleaning)	ACETONE-CLEANED BP (Acetone suspension & Filtration with Cleaning)	METHANOL- CLEANED BP (Methanol suspension & Filtration with Cleaning)
Surface Area (m^2/g)[3]	**45 ± 2**	**217 ± 27** ***(349 ± 10)*** [4]	**348 ± 13** ***(421 ± 6)*** [4]
Pore Size (nm)	**14.9 ± 0.0**	**9.1 ± 0.4** ***(9.8 ± 0.1)*** [4]	**8.0 ± 0.1** ***(8.6 ± 0.1)*** [4]

- [1] Surface area measurement was repeated 2 times per sample and averaged
- [2] Suspended in 1 % surfactants (i.e., sodium cholate and sodium dodecyl sulfate)
- [3] Determined by Brunauer, Emmett and Teller (BET) theory
- [4] Measured after toluene adsorption isotherm in which 960 min of degassing was performed at 300 ˚C

RESULTS: Adsorption Efficiency

- Toluene Adsorption Isotherm[1]
  - Acetone-cleaned BP (150 cm³/g STP)
  - Methanol-cleaned BP (187 cm³/g STP)





[1] Measurement was performed with one representative sample

RESULTS: Adsorption Efficiency

- Toluene Adsorption Capacity[1]

Fabrication Method	Not-Cleaned BP	Acetone-Cleaned BP	Methanol-Cleaned BP
Adsorption Quantity (cm^3 toluene /g BP at STP)	–[2]	**150**	**187**
Adsorption Capacity[3] (cm^3 toluene / g BP)	–[2]	**0.72**	**0.89**
Adsorption Capacity (mg toluene / g BP)	–[2]	**618**	**770**

- [1] Measurement was repeated 2 times and averaged
- [2] Data was not included because of considerably lower surface area
- [3] Conversion of gas volume at STP to liquid volume at analytical temperature using a toluene density conversion factor of 0.0047677

Conclusion/ Future Work

- Cleaning process was necessary for this material (i.e., arc discharge SWNT solution) and methanol cleaning fabrication method was the most efficient
- Toluene adsorption capacity was proportional to surface area
- Increase surface area by fabrication (e.g., annealing)
- Examine other types of sorbents (e.g., HiPco (high pressure CO) SWNTs, carbonaceous particles, etc.)
- Test PhotoThermal Desorption (PTD) efficiency of the samples
- Build a passive sampler unit for photothermal desorption

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