Metal-doped carbon aerogels prepared from oil shale by-products

Kristiina Kreek

Tallinn University of Technology

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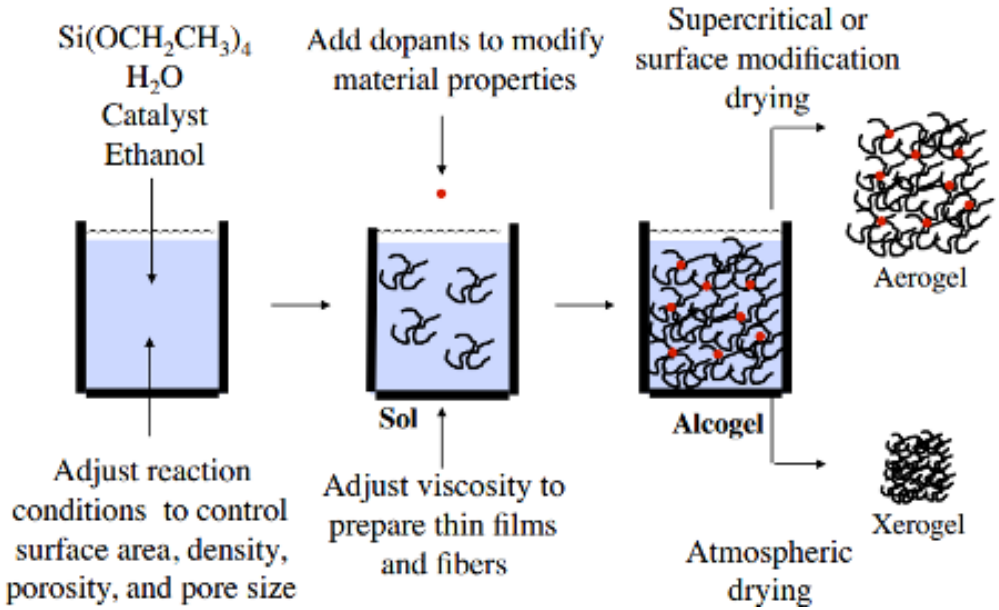
Introduction

- Aerogels are porous materials which are derived from gel by replacing the liquid component with gas, without collapsing the gel solid network [1]



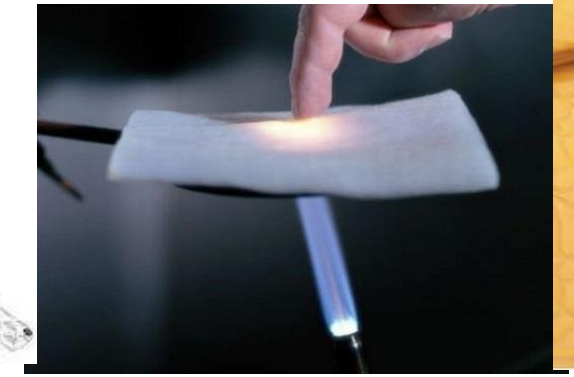
[1] S.S. Kistler. Coherent Expanded Aerogels and Jellies - *Nature*, 1931, 127, 741.

Properties and applications

- Properties:
  - low-density (usually 0,020 g/cm^3 or higher)
  - typically 95%-99% air, open-porous (pore sizes from 1-100 nm)
  - low thermal conductivity (as low as 0,016 W/m*K)
  - high specific surface area (up to 3200 m^2/g, carbon aerogel)

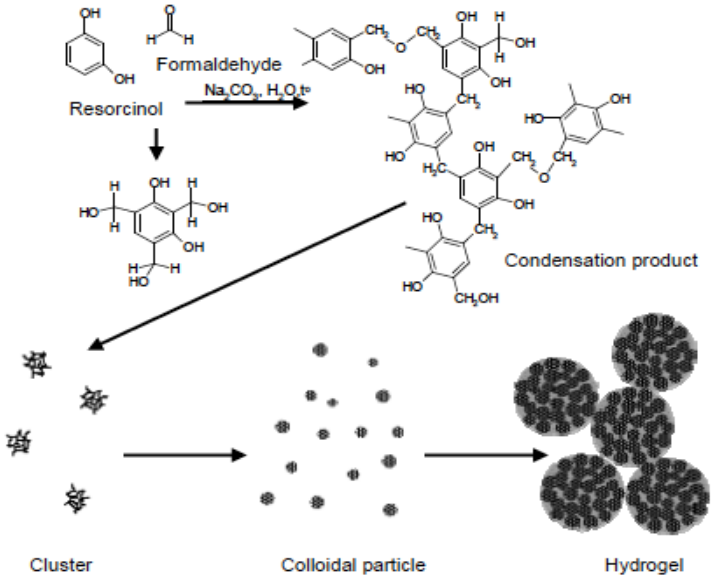
•Applications:

thermal insulators, adsorbent, desiccant, filtering, electrode materials, drug delivery system, platforms for chemical sensors etc.



Organic and carbon aerogels

- Resorcinol-formaldehyde organic aerogels are the most studied



Carbon aerogels are obtained by pyrolysing organic aerogels

Carbon aerogel properties: consist almost entirely of carbon, have high surface areas (up to 3200 m^2/g), continous porous structure and can be electrically conductive

Applications: catalyst support, adsorbent, materials for electrodes

5-methyresorcinol as a local alternative

- Produced by extraction from oil shale retort liquor
- 5-methylresorcinol (MR) is an appropriate compound for preparing organic aerogels [2].
  - faster gelation times than resorcinol
  - gelation at room temperature
- Organic aerogels prepared by using MR as a precursor, have
  - densities as low as 0.1 g/cm^3
  - surface areas higher than 350 m^2/g.
  - carbon aerogel surface areas up to 2000 m^2/g

[2] F. Perez-Caballero, A.-L. Peikolainen, M. Uibu, R. Kuusik, O. Volobujeva, M. Koel. Preparation of carbon aerogel from 5-methylresorcinol-formaldehyde gels - *Microporous and Mesoporous Materials*, 2008, 108, 230-236.

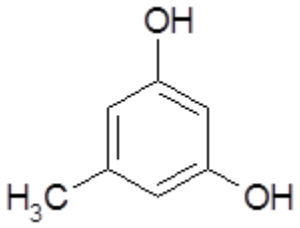
Metal-doped aerogels

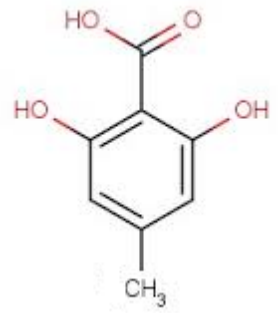
- Doping carbon aerogels with metals enables to modify the structure of the materials, catalytic activity and electrical conductivity.
- Methods that have been used for doping aerogels with metals can be divided roughly into three categories [3]:
  - Adding the metal precursor before gelation into the sol
  - The ion-exchange method
  - Immersing aerogels in metal precursors

[3] C. Moreno-Castilla, F.J. Maldonado-Hódar. Carbon aerogels for catalysis applications - *Carbon*, 2004, 43, 3, 455-465.

My research

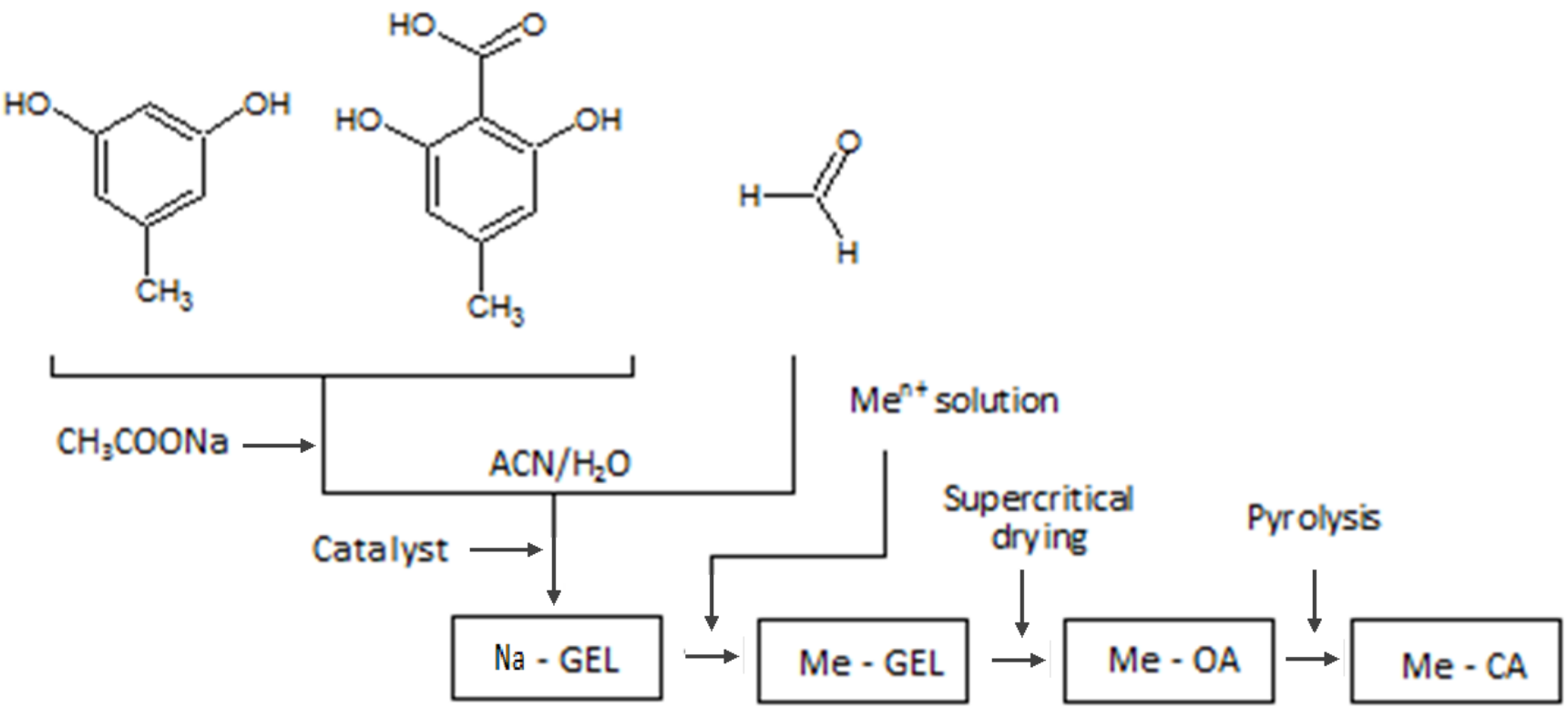
- Preparation of metal-doped carbon aerogels from 5-methylresorcinol (MR) and 2,6-dihydroxy-4-methylbenzoic acid (dHMBA).
- Metal ions are introduced through the ion-exchange process and reduced during pyrolysis.
- Testing of prepared aerogels for new applications





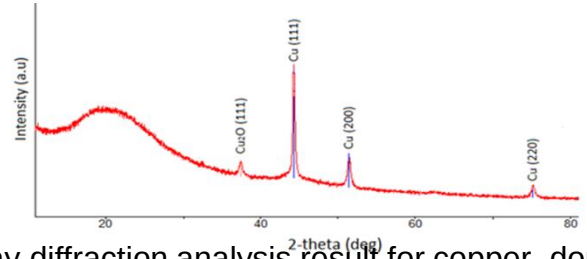
Copper-doped carbon aerogels

The method for preparation



Data on CA-s with metals

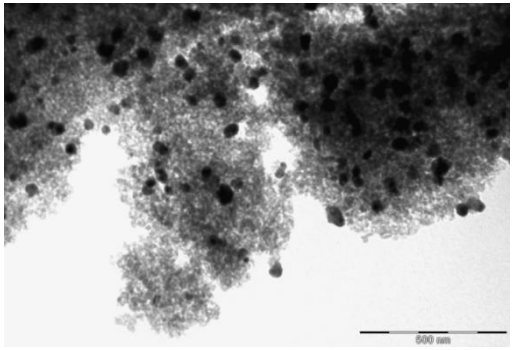
- Surface areas of aerogels are up to 520 m^2/g and pore volumes up to 610 mm^3/g.
- Metal ions are reduced during carbon aerogel preparation.



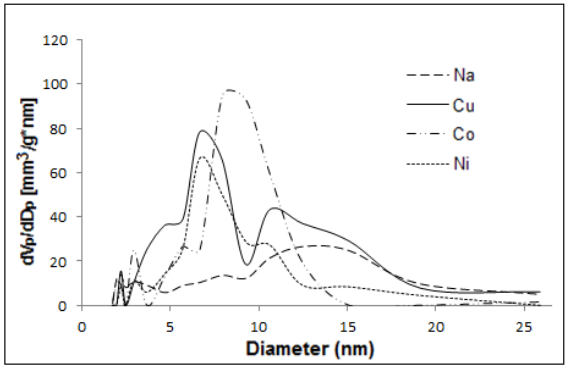
X-ray diffraction analysis result for copper–doped carbon aerogel

- By changing the molar ratio of MR and dHMBA, the metal content can be varied from 1% to 20%, depending on the metal used.

- Carbon aerogels contain metal nanoparticles.



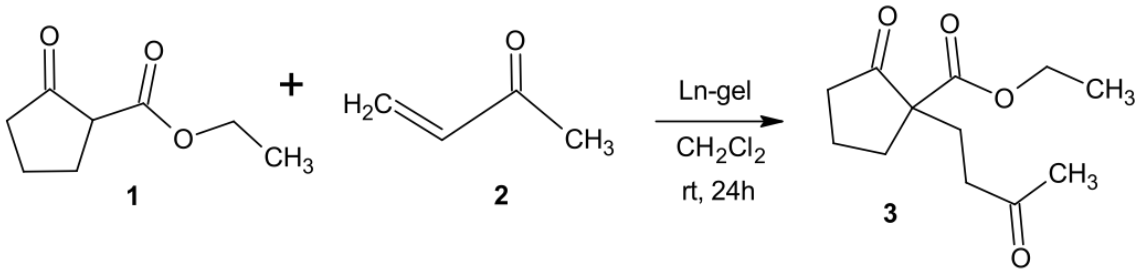
Transmission electron microscope picture of a cobalt-doped carbon aerogel.



Pore size distribution analysis results

Applications for prepared materials

- Catalyst carriers
  - We have tested aerogels doped with lanthanides



- Chemical sensors printed on paper
  - Can be used for detecting toxic chemicals
  - Carbon surface adsorbs many organic species onto surface, so measurements based on an electrical response can be conducted.
  - Paper is cheap and biodegradable

Conclusion

- Estonian oil shale production by-products 5-methylresorcinol and 2,6-dihydroxy-4-methyl benzoic acid are good precursors for organic and carbon aerogel preparation.
- The method enables to prepare carbon aerogels with high surface areas and to easily control the metal content in aerogels.
- Prepared aerogels are promising materials for several applications.

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