

REMOVAL OF CARBONACEOUS CONTAMINANTS FROM SILICA AEROGEL. Hui-Ping Huang, I. Gilmour and C.T. Pillinger, Planetary Sciences Unit, Department of Earth Sciences, Open University, Milton Keynes, UK. M.E. Zolensky, Johnson Space Center, Houston, Texas, USA.

Capture of micrometeorite material from low Earth orbit or dust grains around active comets for return to terrestrial laboratories, capable of practicing the most up to date techniques of chemical isotopic and mineralogical analysis, will greatly enhance our knowledge of primitive material in the solar system. The next generation of space launched cosmic dust collectors will undoubtedly include extremely low density target materials such as silica aerogel as the decelerating and arresting medium. This material has been found to be clean from the point of view of inorganic elements (1, 2) and is thus acceptable for the purpose of harvesting grains to be studied by, for example PIXE, INAA or SXRF. On the contrary however the process used in making aerogel leaves substantial carbon and hydrogen containing residues (3, 4) which would negate their suitability for collection and subsequent investigation of the very important CHON particles. Attempts to precondition aerogel by solvent extraction or heating at 500°C and 750°C in air for 24 hours or under a vacuum of 2×10^{-7} torr at 260°C were largely ineffective except that pyrolysis did reduce volatile species. In this investigation we have examined the use of supercritical fluids for the purpose of extracting organic residues. The logic of the new approach is that beyond the supercritical point a substance has the solvating properties of a liquid but the viscosity characteristics of a gas. For example carbon dioxide becomes supercritical at a pressure of 73 atmospheres and a temperature of 31°C; in consequence it can transform to a very powerful and ultraclean solvent. It can dissolve organic matter from low molecular weight up to molecules containing 90 carbon atoms. On release of pressure the fluid reverts to a gas which can easily be pumped away and removed from the substrate being extracted.

Preliminary trials using supercritical carbon dioxide conducted with 0.12g/cm³ aerogel (the lowest density material and hence presumably the most open structured available) show considerable promise. Our previous analyses (4) of this substrate and more recent results by both bulk and stepped combustion suggest a relatively low but still unacceptable carbon content of 0.18 to 0.27 wt %. Approximately 85% of this contamination is liberated by burning aerogel at 600°C attesting to the organic nature of the contaminant carbon. The Table shows carbon yields following various treatments with supercritical CO₂. It can easily be seen that the total carbon can be reduced without too much effort by at least a factor of ten.

In two analyses we tried modifying the supercritical CO₂ by the addition of methanol, a standard technique to increase polarity. As can be seen in the Table this greatly added to the carbon content demonstrating how difficult it is to eliminate solvents from the aerogel structure. During the most successful experiment (#5) we monitored the weight loss of the aerogel during temperature increase (Fig.). A total reduction in sample mass of nearly 6% was observed, much of it (75%) at the minimum temperature used. It appears that CO₂ at pressure is able to flush the sample of water at practically ambient temperatures assuming that the weight loss is not simply attributable to particulate matter becoming suspended in the mobile fluid phase. The latter interpretation does not seem likely otherwise capillaries in the pressure cell of the extraction apparatus would inevitably become blocked.

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Table.Carbon yields from 0.12 g/cm³ silica aerogel following various treatments with supercritical CO₂.

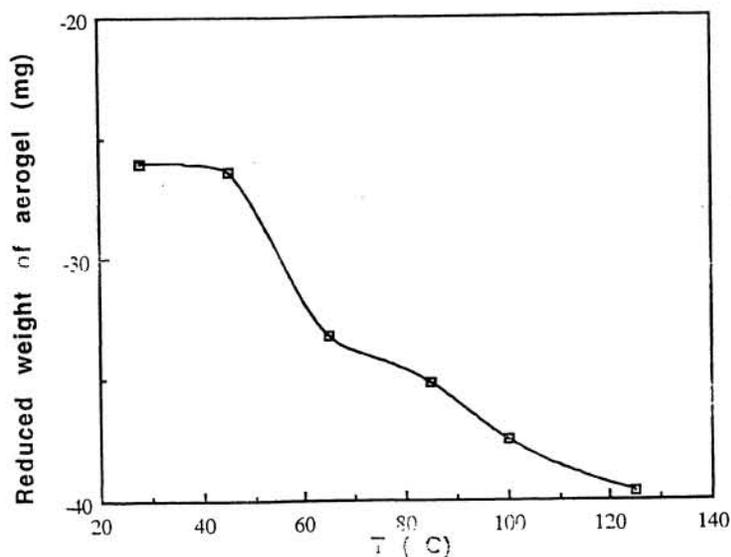
| Ex. No. | P(psi) | T(°C) | Modifier | Sample Wt(mg) | Carbon (%) |
|---------|--------|-------|----------|---------------|------------|
| 1 | - | - | - | 27.8 | 0.27 |
| 2 | 3000 | 50 | - | 48.4 | 0.20 |
| 3 | 4000 | 46 | - | 38.0 | 0.080 |
| 4 | 4000 | 85 | - | 27.6 | 0.095 |
| 5 | 4000 | 150 | - | 32.2 | 0.014 |
| 6 | 4000 | 46 | 1% MeOH | 70.4 | 0.70 |
| 7 | 3000 | 55 | 5% MeOH | 56.3 | 2.80 |

In this initial investigation it is quite likely that we have not yet identified the optimum conditions for cleansing aerogel. We wish to explore the efficacy of SFE with the more dense forms of the substrate richer in carbon. Non-carbon containing supercritical fluids need to be tried. For the method to be suitable for preparing aerogel targets for space launch, a facility capable of accommodating relatively large slabs of the material will need to be constructed.

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References:

1. G. Flynn and S.R. Sutton (1990) LPSC XXI, 371 - 372.
2. M.E. Zolensky *et al.* (1990) LPSC XXI, 1381 - 1382.
3. C.P. Hartmetz *et al.* (1990) LPSC XXI, 463 - 464.
4. J.E. Gibson *et al.* (1991). LPSC XXII, 441 - 442.



SFE operation: CO₂ as mobile phase, 4000 psi, 125 - 23 °C.
 The initial weight of aerogel for extraction: 595.75 mg.
 The final reduction of weight after SFE: 4.38%.
 The carbon composition in the final extract: 0.014%.