Phase Behavior of Polystyrene in Dimethyl Ether, Dimethyl Ether + Carbondioxide

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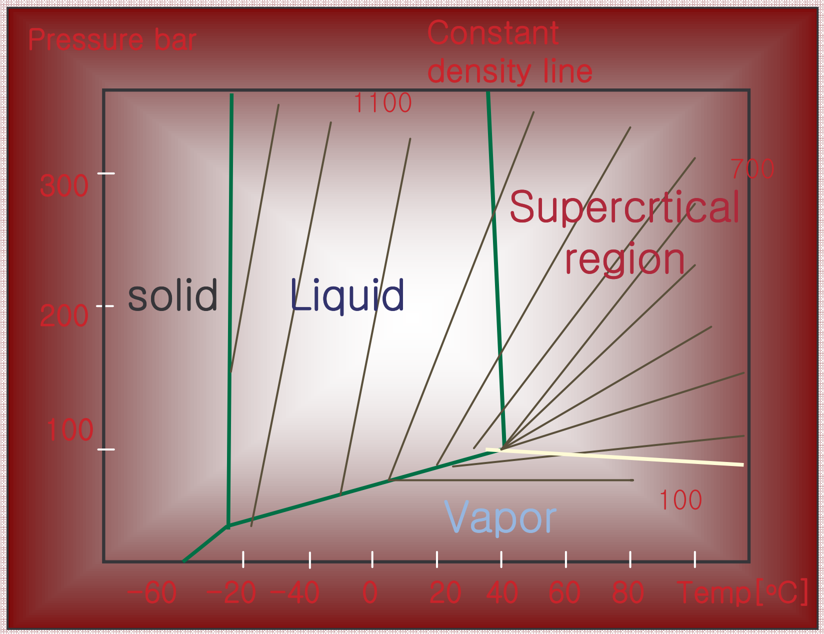
Supercritical Fluid and Green Processing Lab

Introduction – What is the supercritical fluid?

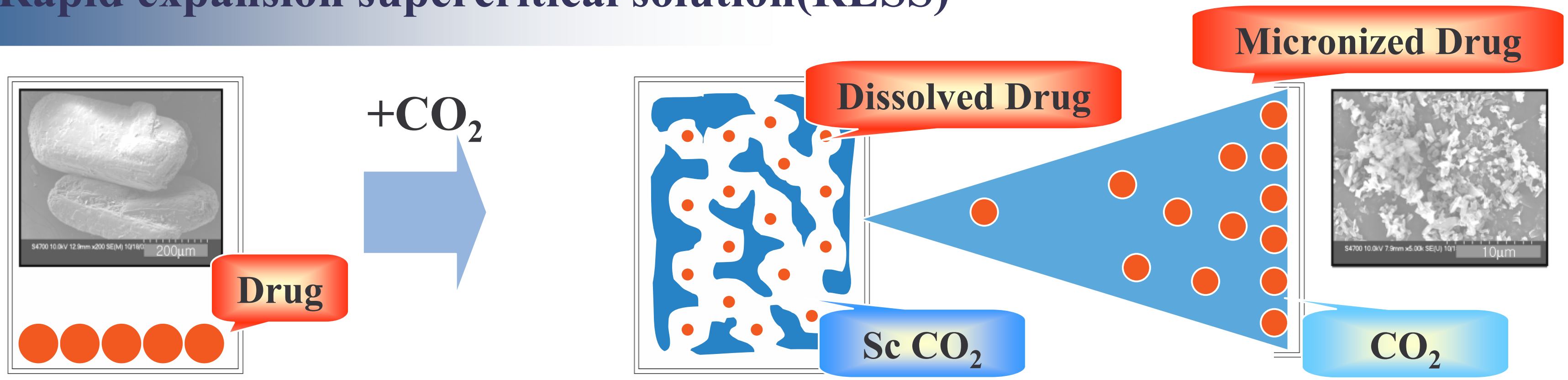
- Noncoxic, chemically inert, cheap, highly volatile, nonflammable
- Liquidlike dissolving power
- Gaslike transport property
- Potentially recyclable fluid
- Attractive as a solvent
- Application
  -Extraction
  -Dry
  -Separation
  -Synthesis reaction
  -Particle design



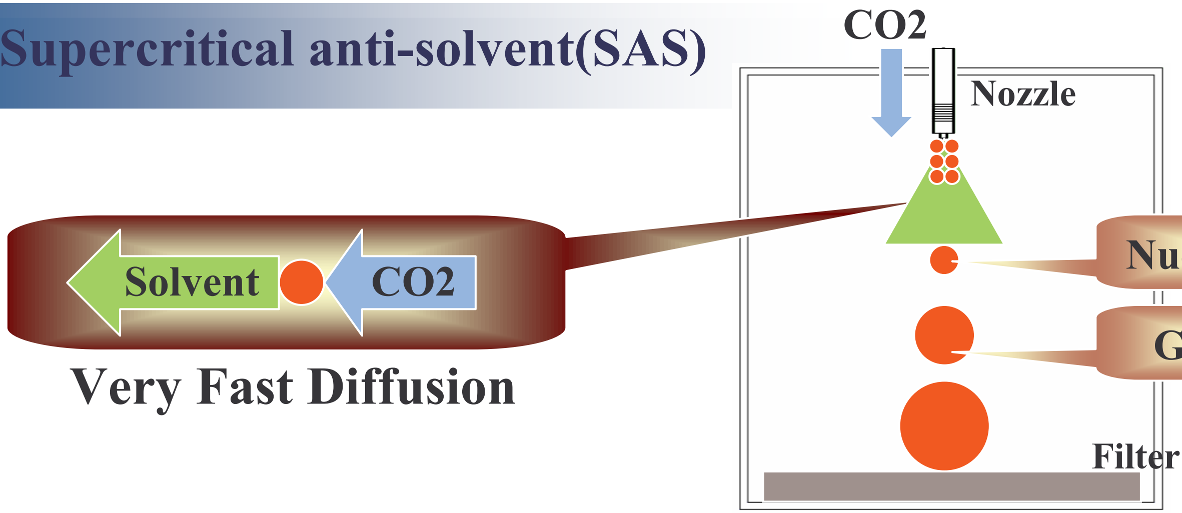
Phase diagram of CO_2

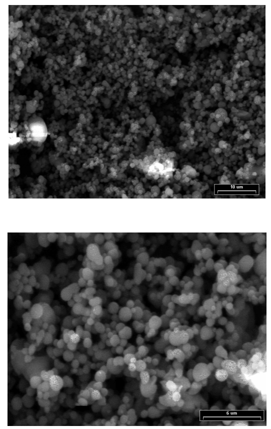
Design of particles using supercritical fluids

Rapid expansion supercritical solution(RESS)

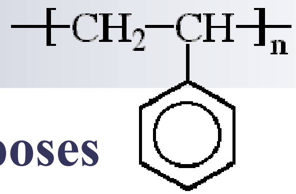


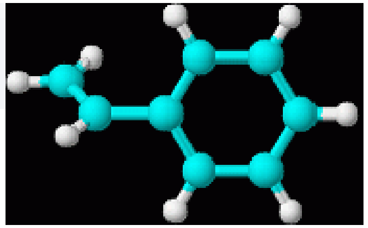
Supercritical anti-solvent(SAS)





Polystyrene

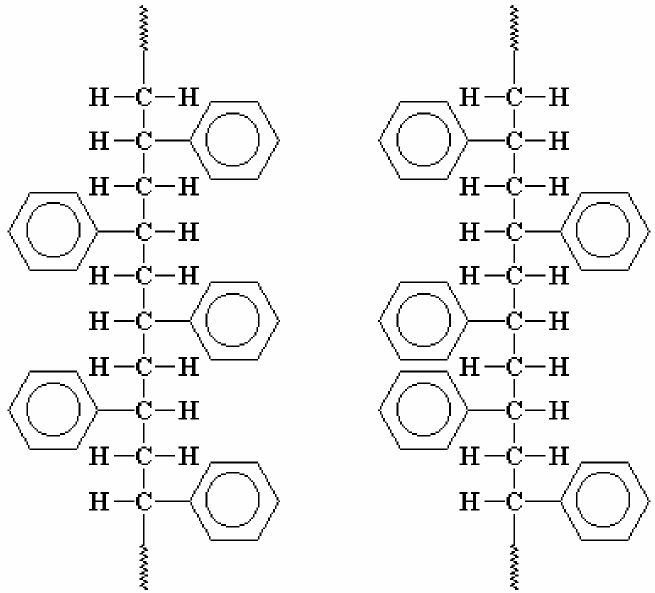




- **Polystyrene is widely used for industrial purposes**

styrene polystyrene

- **Polystyrene is an inexpensive and hard plastic**

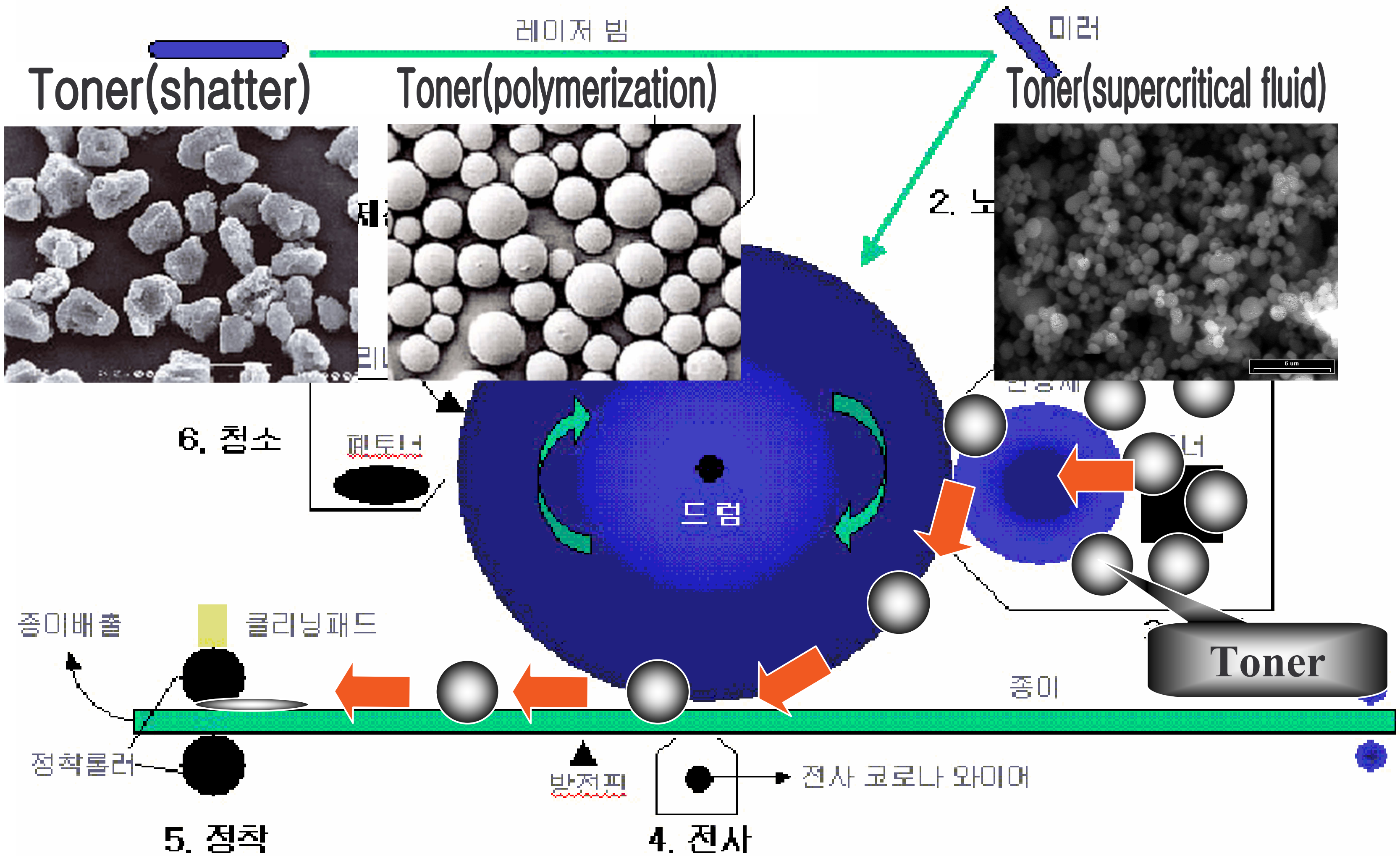


syndiotatic
more expensive

atatic

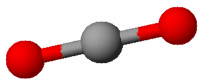
- **-Usage** thermoplastics
  -Monomer styrene
  -Glasstransion temperature 373.15K
  -Melting temperature 543.15K(syndiotatic)
  -Polymerization free radical chain polymerization(atatic)
  - Ziglar-Natta Polymerization(syndiotatic)

Toner



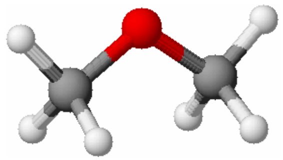
레이저 빔
미러
Toner(shatter)
Toner(polymerization)
Toner(supercritical fluid)
2.
6. 청소
드럼
종이배출
클리닝패드
종이
Toner
정착롤러
받침피
전사 코로나 와이어
5. 정착
4. 전사

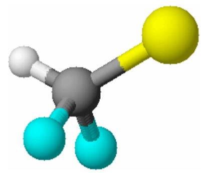
Dimethyl Ether(DME), HCFC-22,CO_2



CO2

Polar molecule





Nonpolar molecule

DME HCFC-22

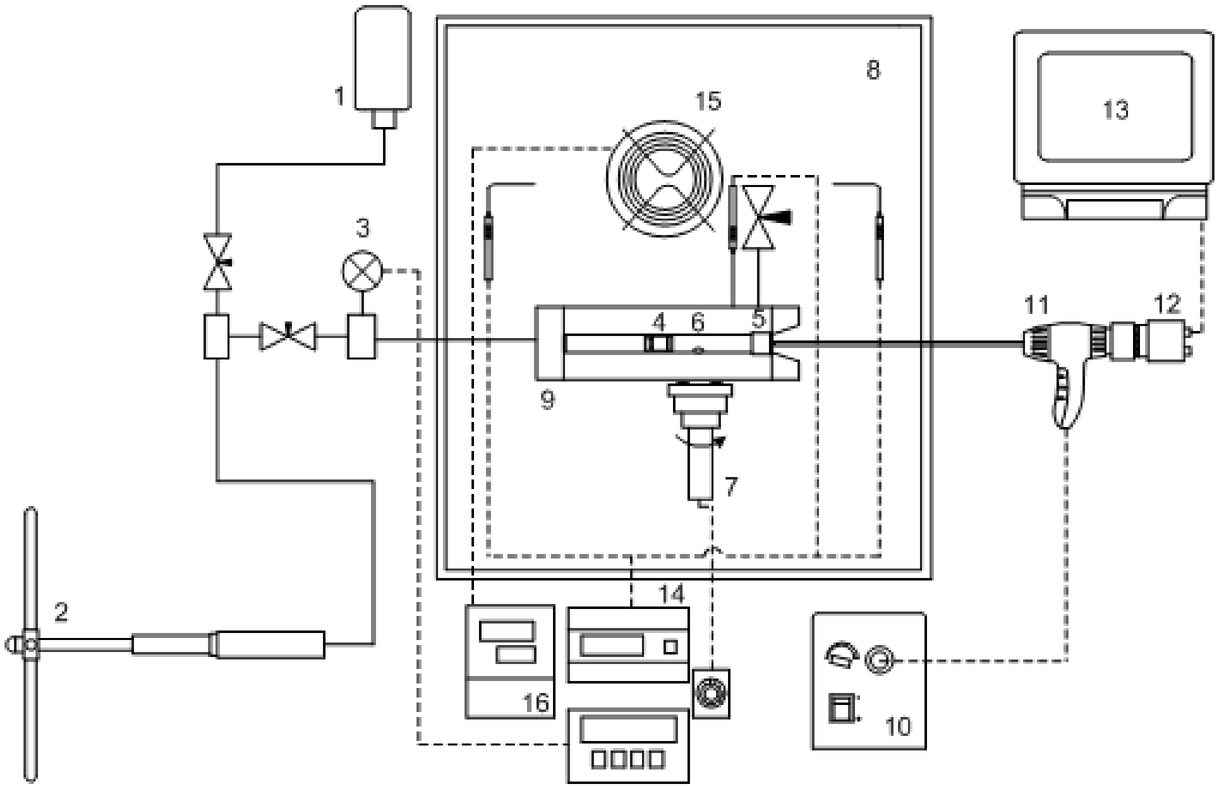
Dipole monents

	μ/D		μ/D
H_2O	1.85	DME	1.3
CH_3OH	1.71	HCl	1.08
C_2H_5OH	1.69	$CHCl_3$	1.01
CH_2Cl_2	1.57	HBr	0.80
NH_3	1.47	HI	0.42
HCFC-22	1.4	CO_2	0

Physical properties of solvents

Solvent	Chemical formula	M.W.	Tc/K	Pc/MPa
Dimethylether (DME)	CH_3OCH_3	46.06	400.00	5.24
Chlorodifluoromethane (HCFC-22)	$CHClF_2$	84.46	369.30	4.97
Carbon dioxide	CO_2	44.01	304.18	7.38

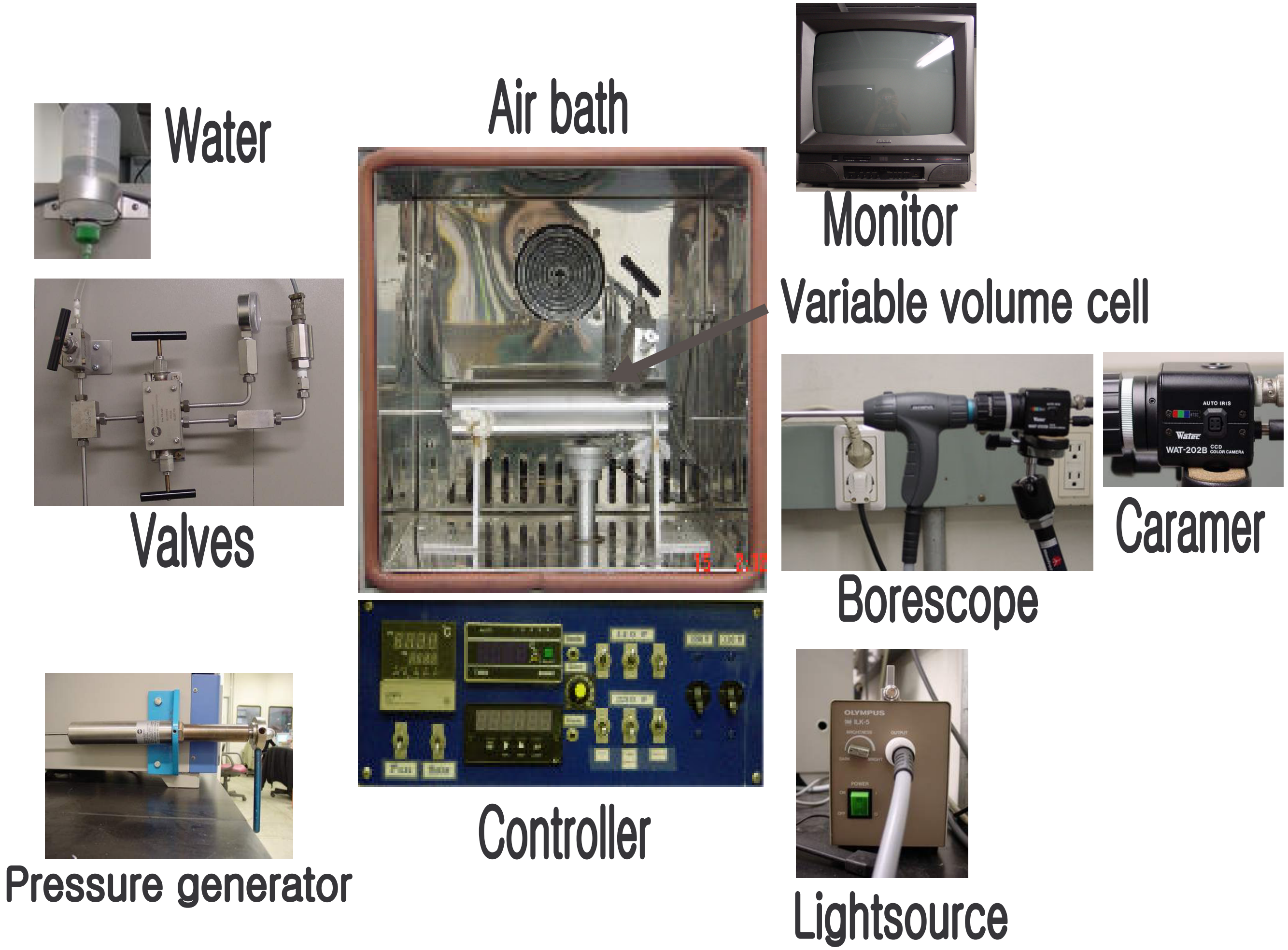
Apparatus – Schematic diagram



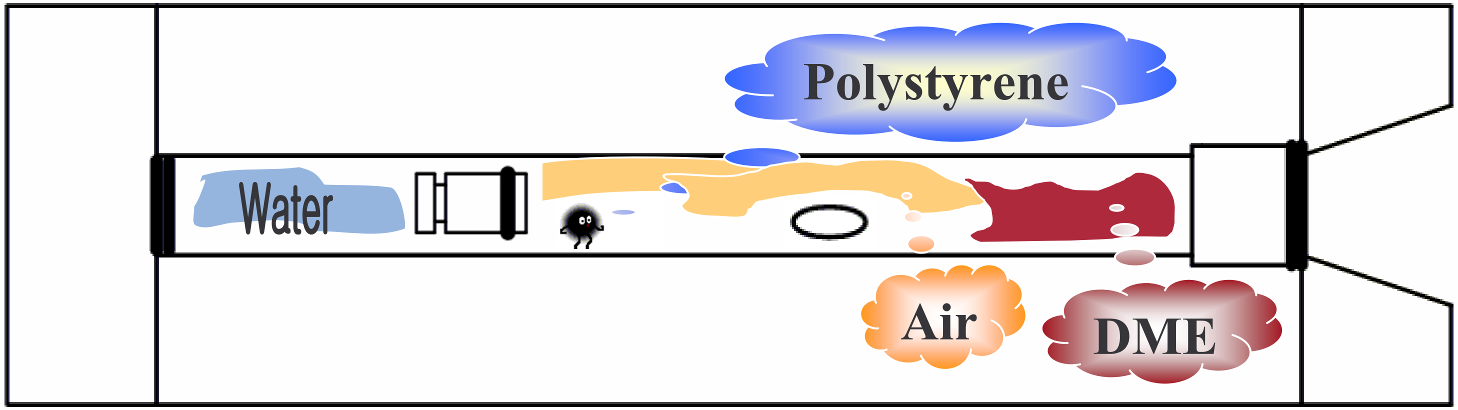
Schematic diagram of experimental apparatus

1. Water for pressing
2. Pressure generator
3. Pressure gauge
4. Piston
5. Sapphire window
6. Magnetic bar
7. Stirrer
8. Air bath
9. Variable-volume cell
10. Light source
11. Borescope
12. Camera
13. Monitor
14. Temperature gauge
15. Heater
16. Heating controller

Photography

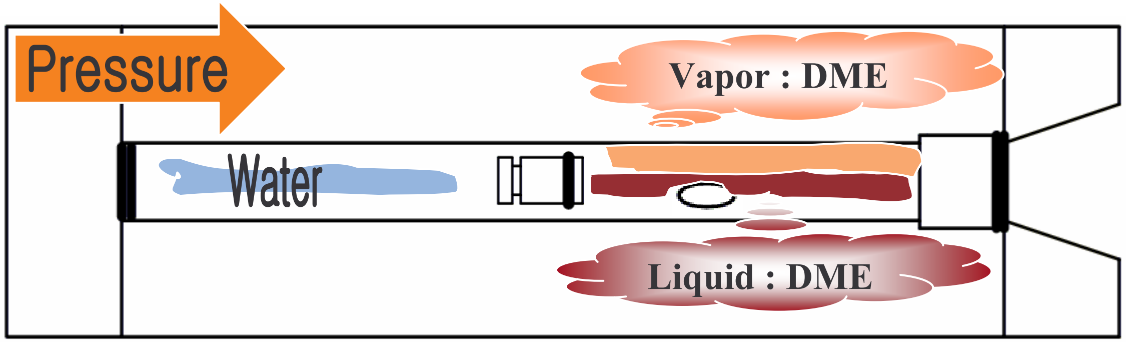


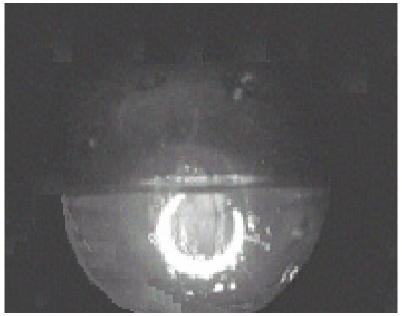
Experimental Procedure



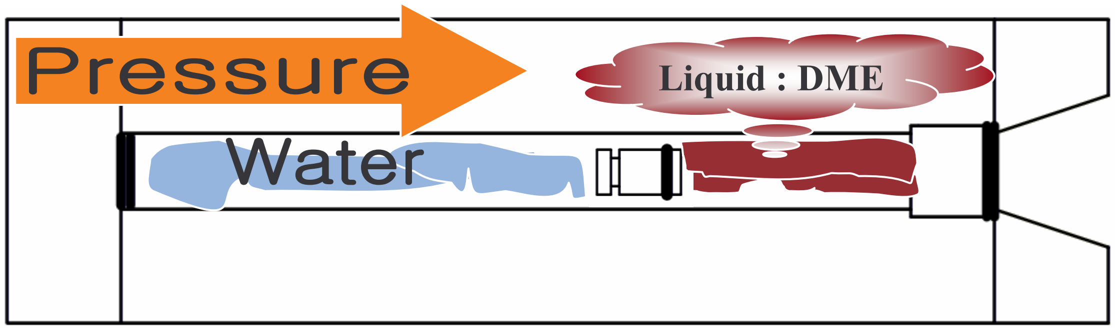
- The cell is charged with a certain amount of polystyrene
- To remove air, the cell is purged more than three times by DME
- A certain amount of DME is charged into the cell

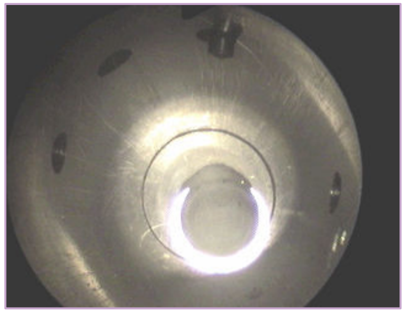
Pressure increase – single phase – Temperature increase





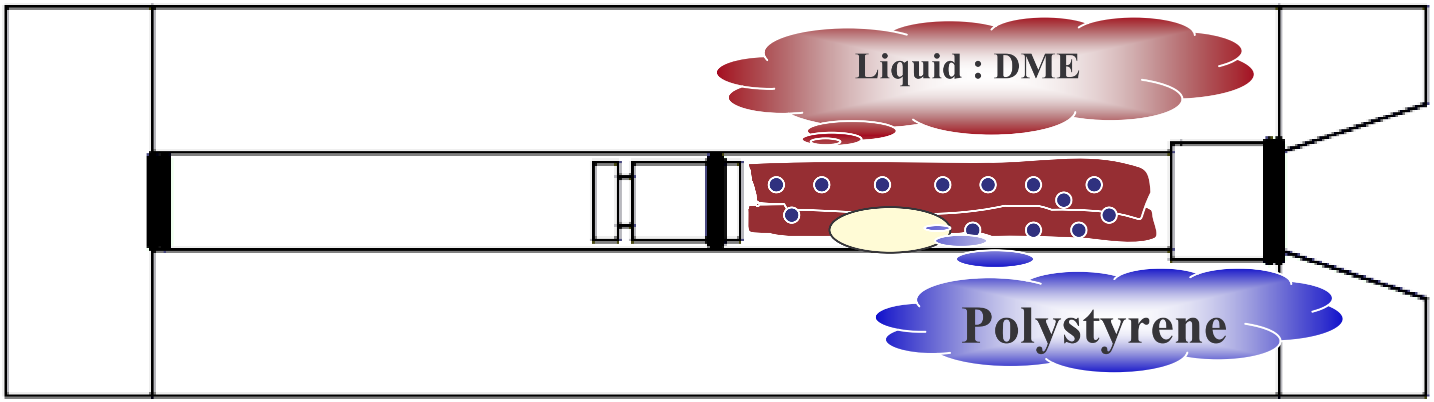
As the pressure increase, the DME finally becomes a single phase



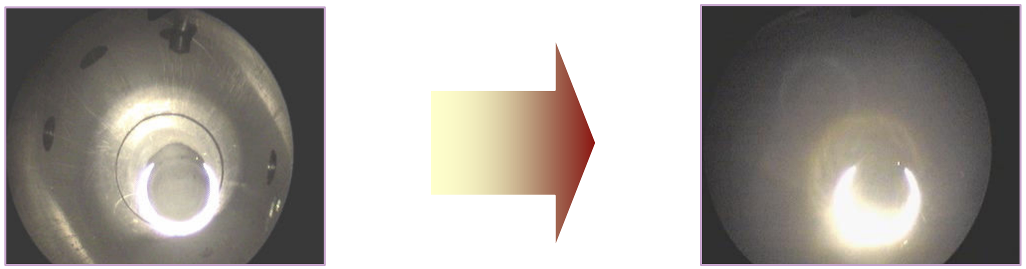


The temperature of the cell is increased by heater

Cloud point measurement



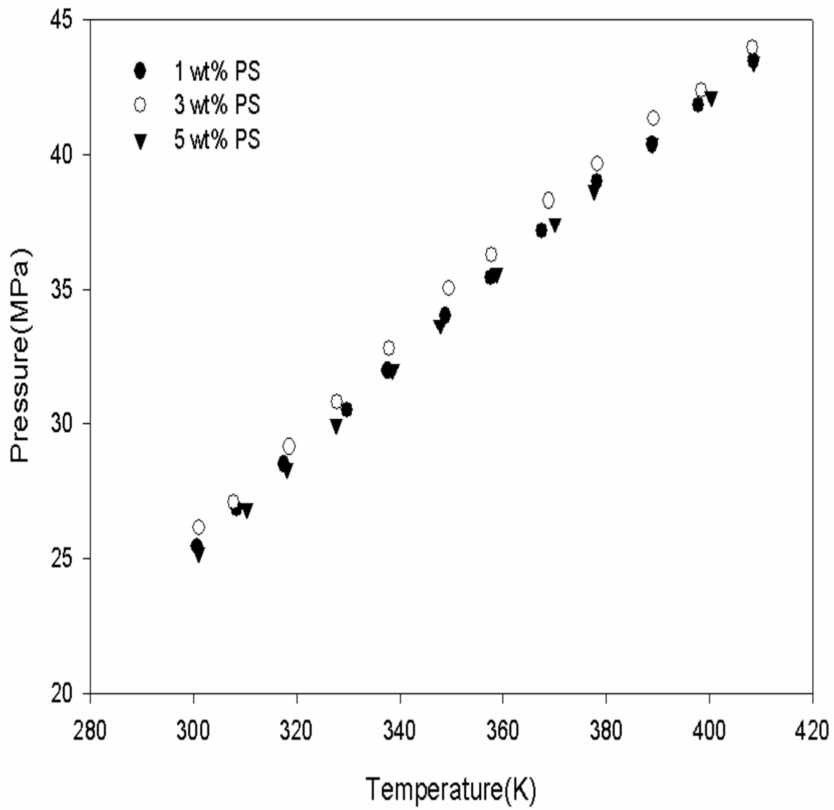
- At each constant temperature, the cloud points were measured By adjusting pressure of the DME by using pressure generator

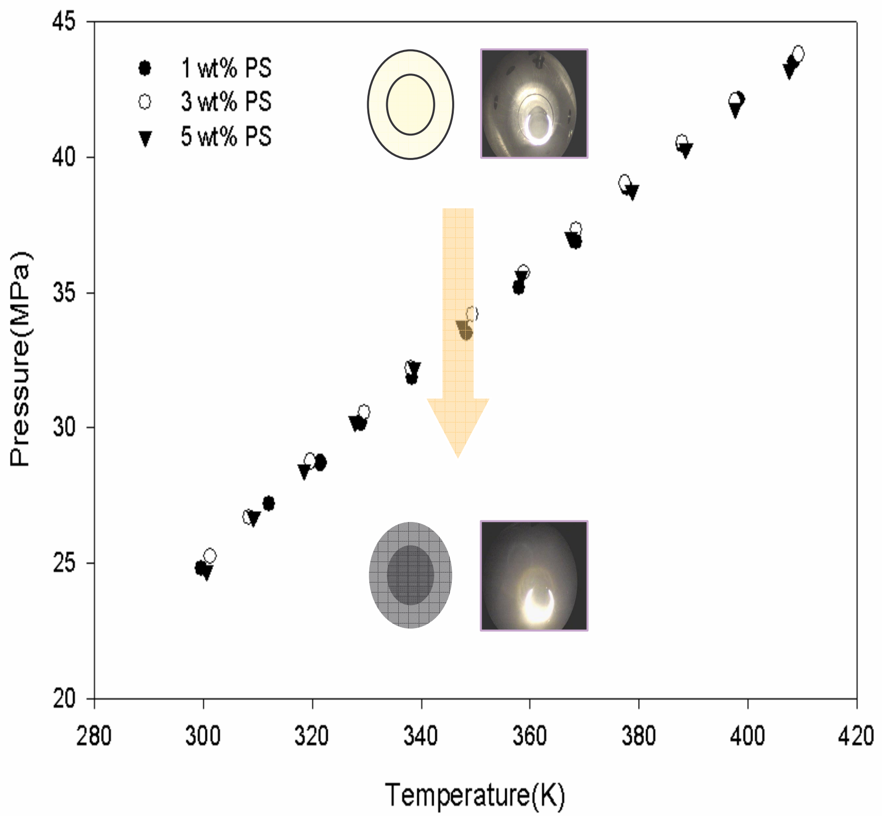


Cloud points

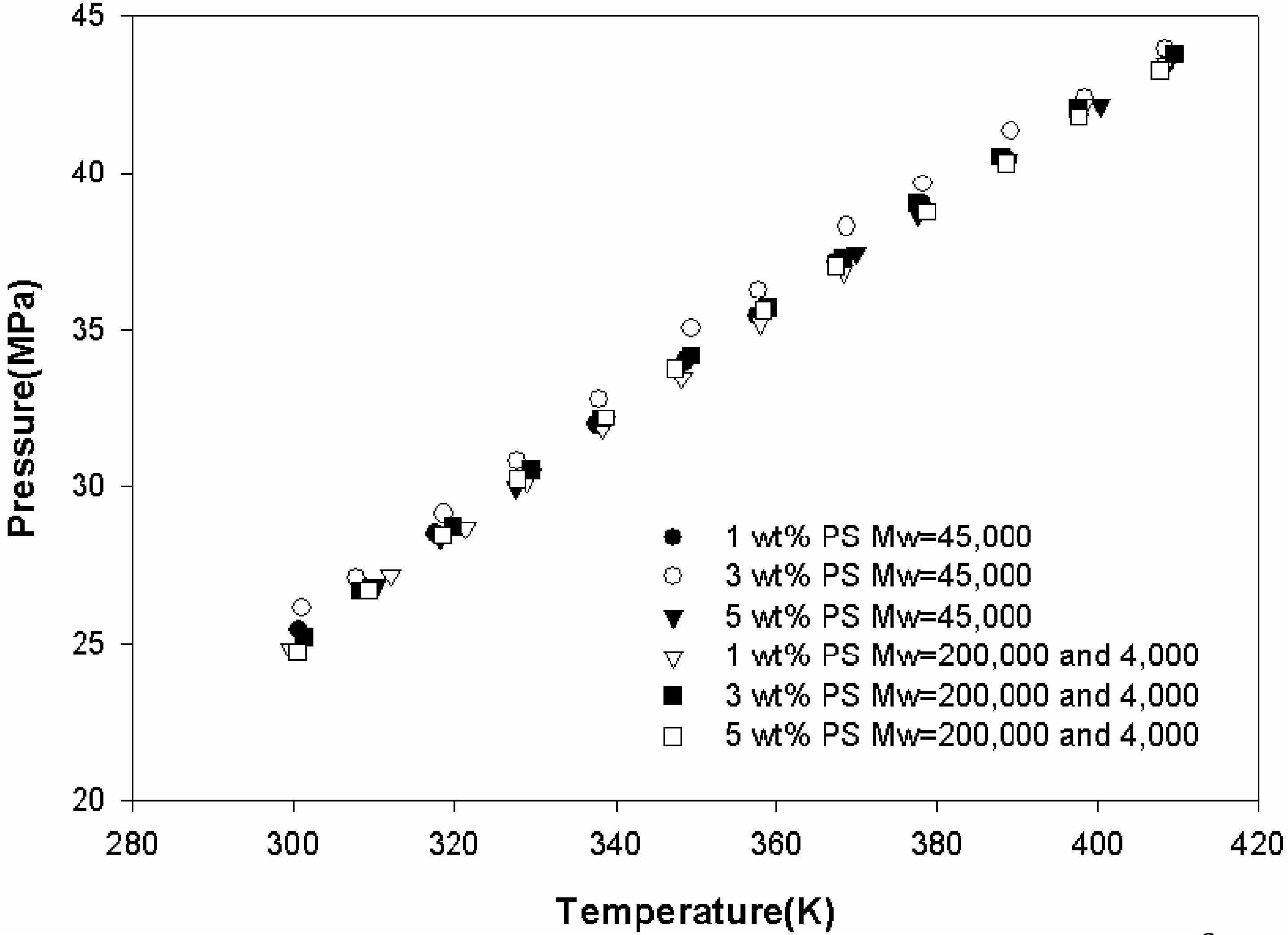
Results & discussion

PS(Mw=45,000)+DME PS(Mw ca.=200,000 and 4,000)+DME



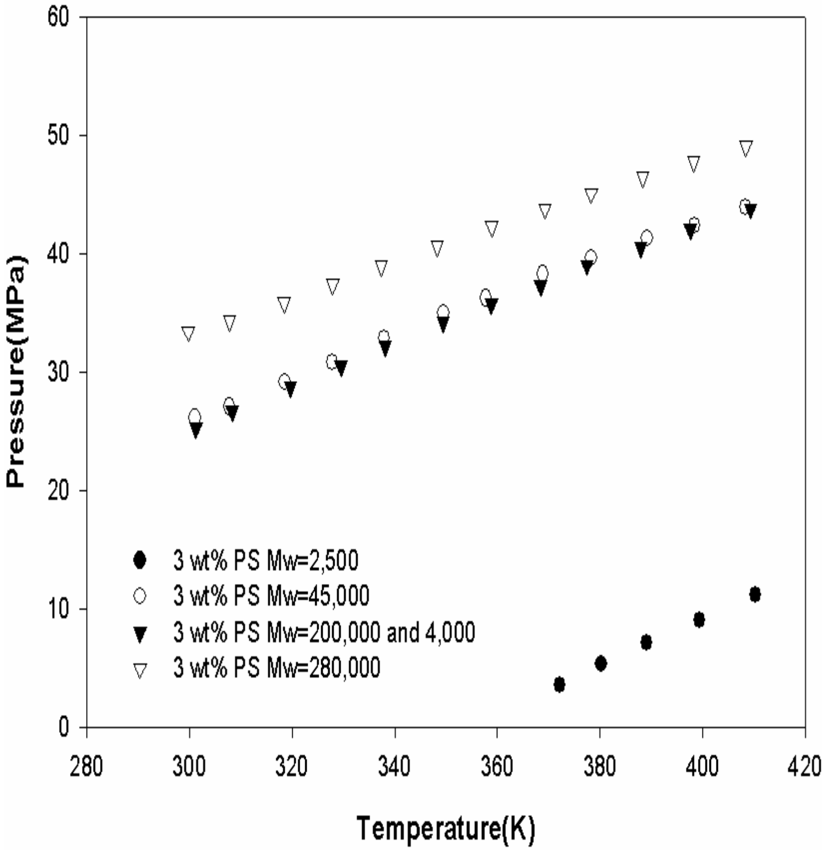


PS(Mw = 45,000) + PS(Mw ca. = 200,000 and 4,000) + DME

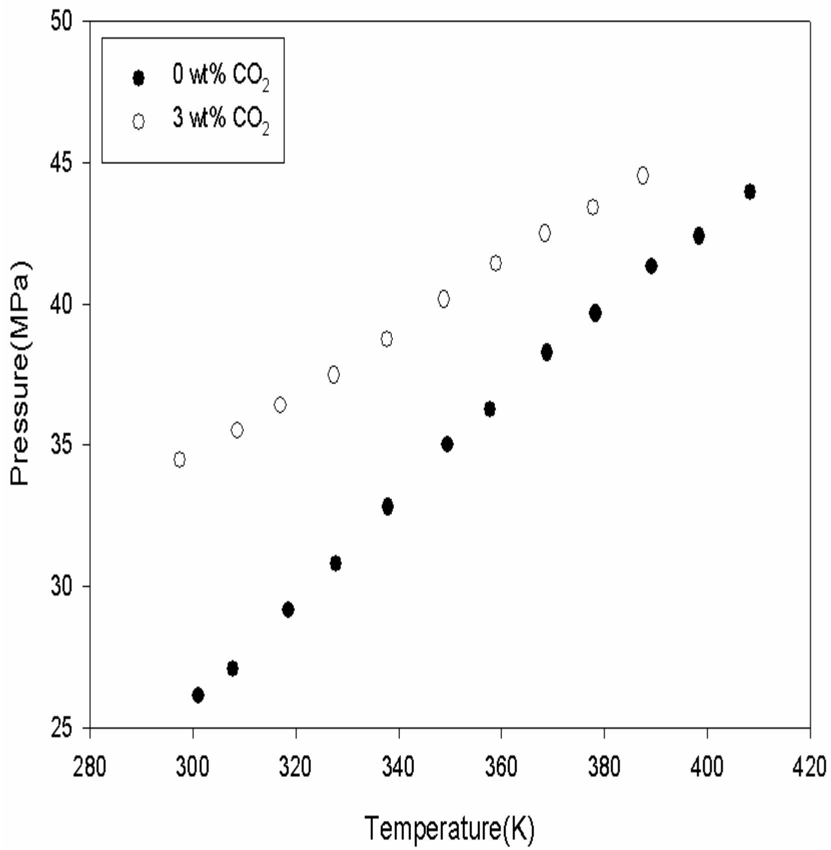


PS(2,500/45,000/20,000 and 4,000/280,000) + DME , PS + DME + CO_2

PS + DME



PS(Mw = 45,000) + DME + CO_2



Conclusions

- The phase behavior of PS in DME, DME+CO_2
- Temperature range from 300 to 410K and pressure up to 150Mpa
- LCST phase behavior was observed
- The cloud point pressure of PS increased proportionally to the amount of CO_2 added at the same temperature
- PS wat not dissolved in DME+CO_2 (13 wt% and 18wt%) even at 410.00K and 50MPa
- CO_2 could be used as an anti-solvent, and the cloud point of PS could be controlled by changing the concentration of CO_2
- PS was not dissolved in HCFC-22 even at 411.13K and 150 MPa