Preparation of Polypropylene Hollow Fiber Membranes for Submerged System

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Requirement of Membranes for Submerged System

1. Chemical and thermal resistances
   for waste water treatment
2. Mechanical strength for handling and air bubbling
3. High porosity and high water flux
4. Durability and stability
5. Less fouling
6. Cost Effective

Polypropylene Hollow Fiber Membranes

Other Applications of Polypropylene Membranes

1. Membrane separation under coarse conditions
2. Medical applications
   - blood oxygenator
   - bacteria filter
   - leukocyte filter
3. Vapor permeation
   - breathing wear
   - diaper film,
   - degasing rinse water in semiconductor industry
4. Battery separator

Methods for Making Polypropylene HF Membranes

- Thermally-induced phase separation (TIPS) process

Procedure
1. Melt blending of polymer and diluent at high temperature
2. Forming into desired shape
3. Phase separation of polymer and diluent by cooling
   Liquid-liquid phase separation –→ cell structure
   Solid-liquid phase separation –→ spherulitic structure
4. Diluent extraction and drying

Advantages
- Wider choice of membrane materials
- Uniform structure
- Easy structure control
- Heat and chemical resistant

Polymer materials lab

- Stretching process

Procedure
1. Stretching the semicrystalline polymer
2. Pore and microfibril formation at amorphous region
3. Heat treatment for relaxation

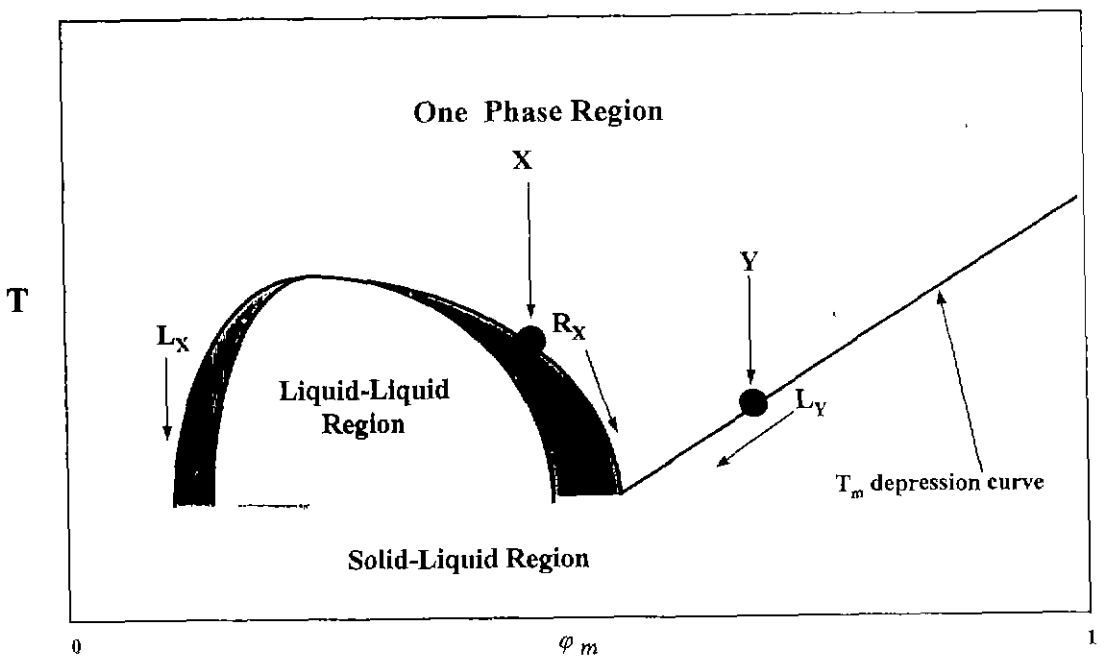
Advantages
Clean process (no chemical)
Uniform structure

- Combination of TIPS and stretching process

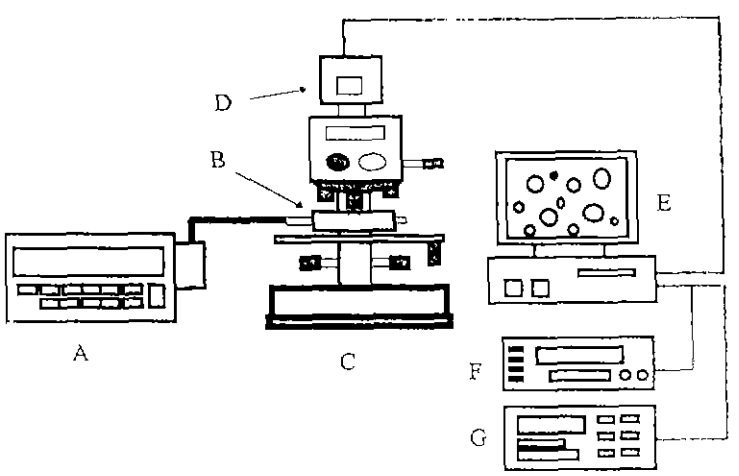
Very high porosity
Uniform structure

Polymer materials lab

Phase diagram for the binary system of polymer and diluent



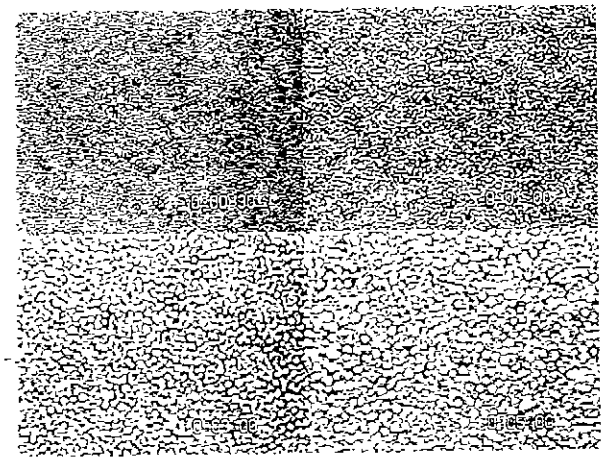
(Polymeric Materials Lab.



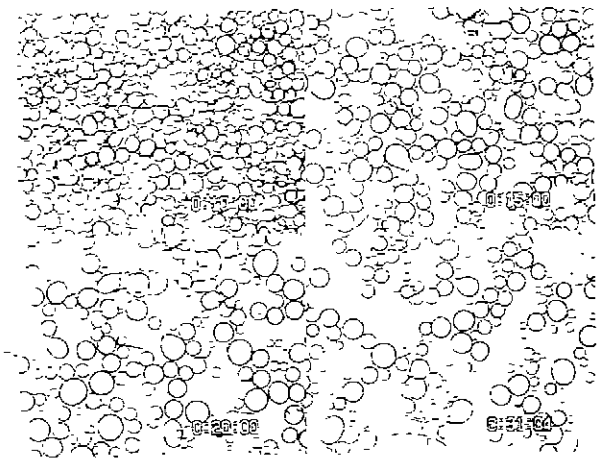
Thermo-optical microscope system

A Central processor B Hot stage, C Optical microscope,
D CCD camera, E Image analyzer, F VCR
G Color video printer

❒ Images of iPP/DPE = 10/90 sample phase separated at 110 °C
(Each holding times is specified on each pictures; hr:min:sec)

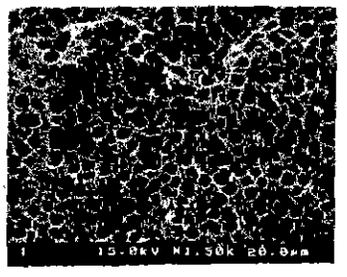


(continued)

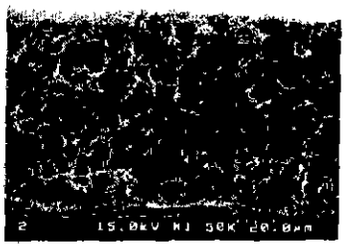


❒ SEM Photographs (for iPP/DPE = 20/80 wt% samples)

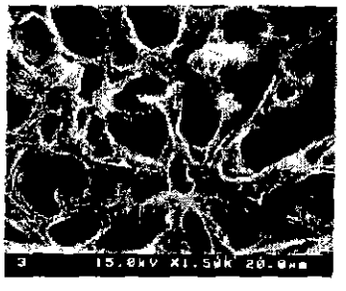
- Holding Time in L-L phase separation region : 10 min



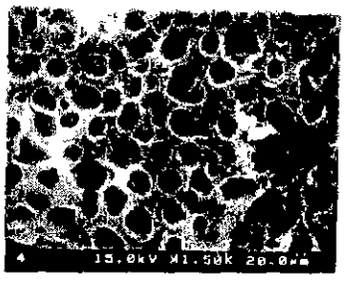
(a) 210 °C → 120 °C



(b) 210 °C → 115 °C



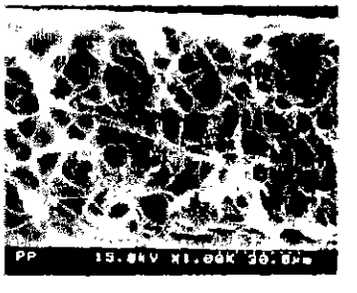
(c) 210 °C → 110 °C



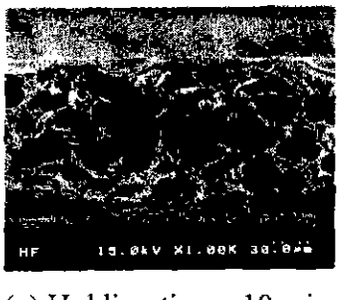
(d) 210 °C → 105 °C

- Quenching Temperature : 110 °C

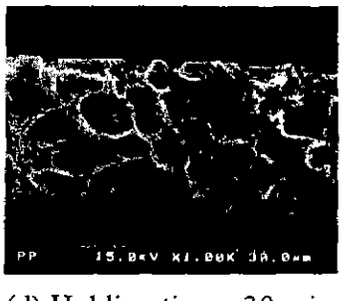
(a) Holding time : 1 min



(b) Holding time : 5 min



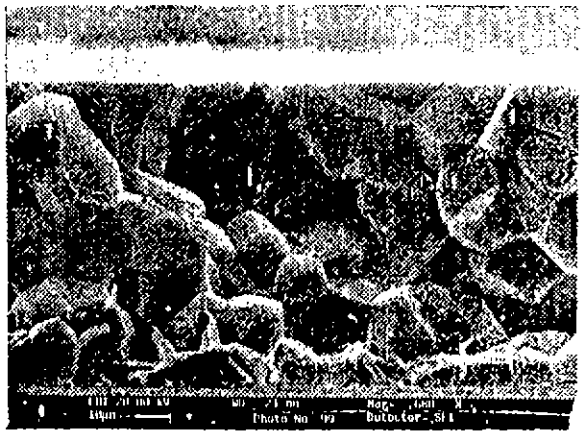
(c) Holding time : 10 min

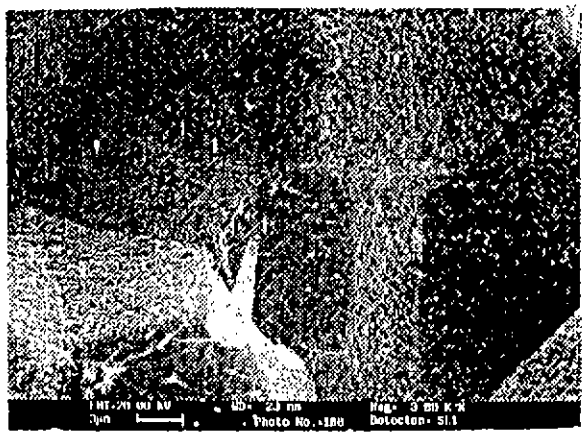


(d) Holding time : 30 min

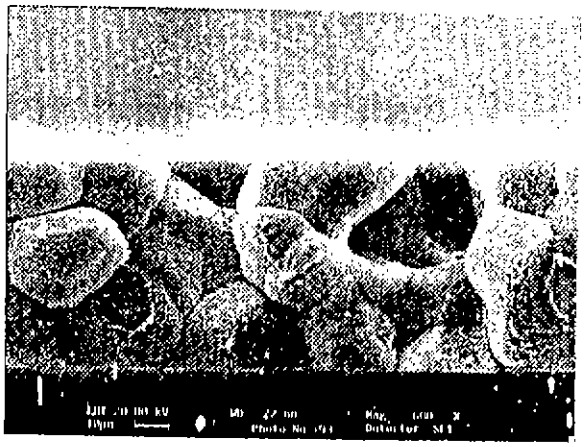
PP 70 wt%

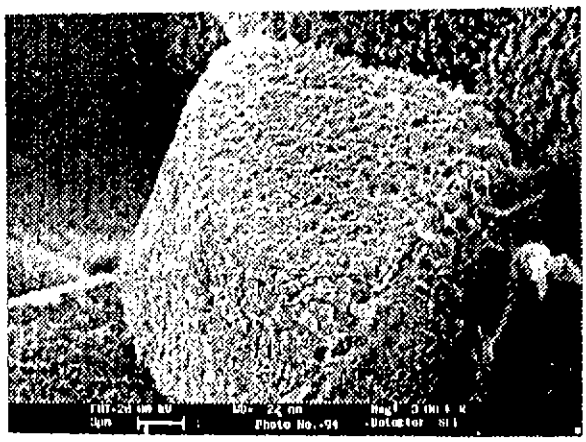
140 ℃



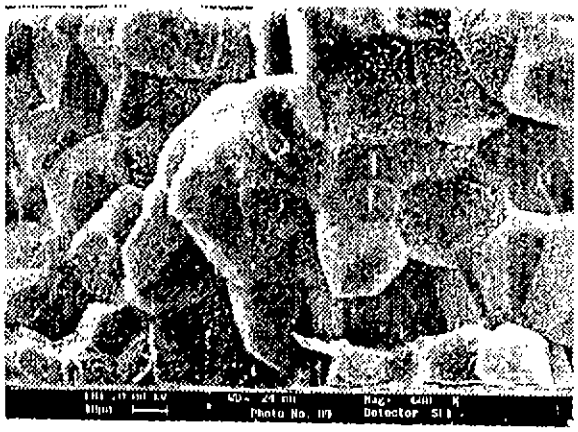


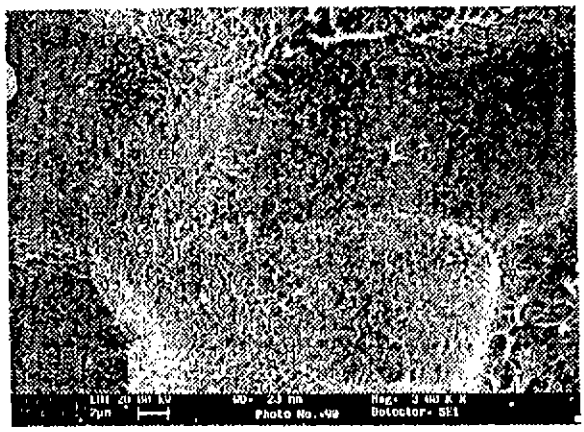
110 ℃



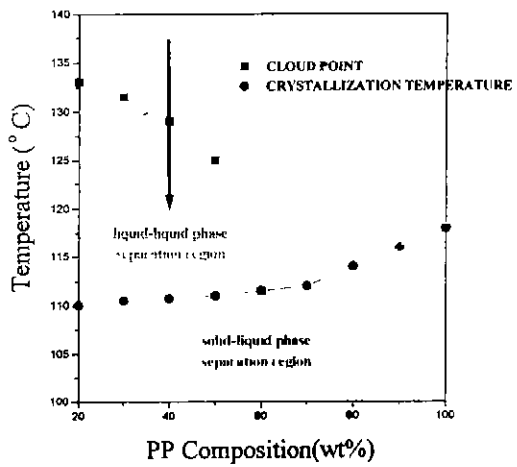


25℃

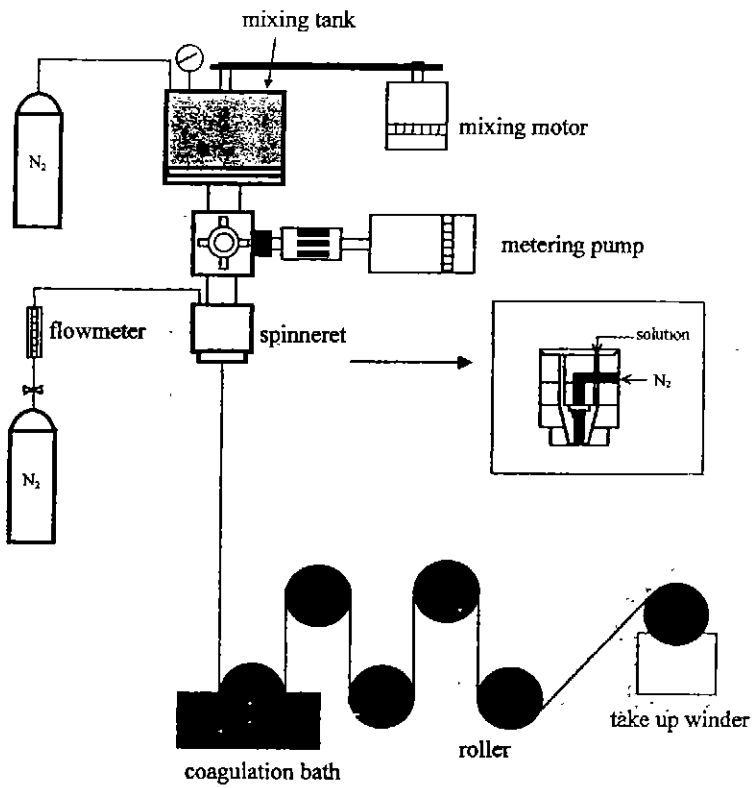




❑ Phase diagram of PP - soybean oil system



❑ **Hollow Fiber Spinning Apparatus**

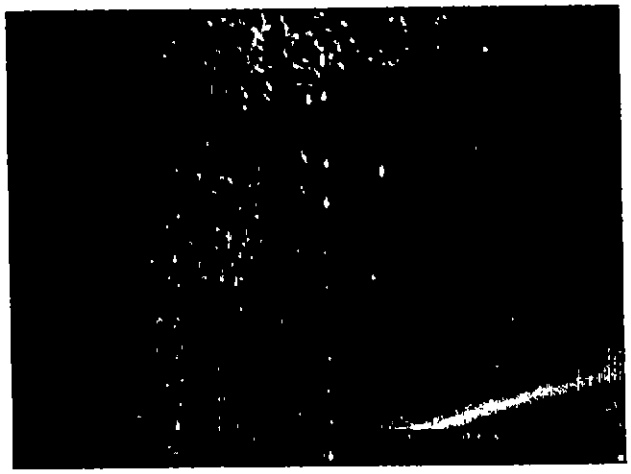


Polymeric Material Lab.

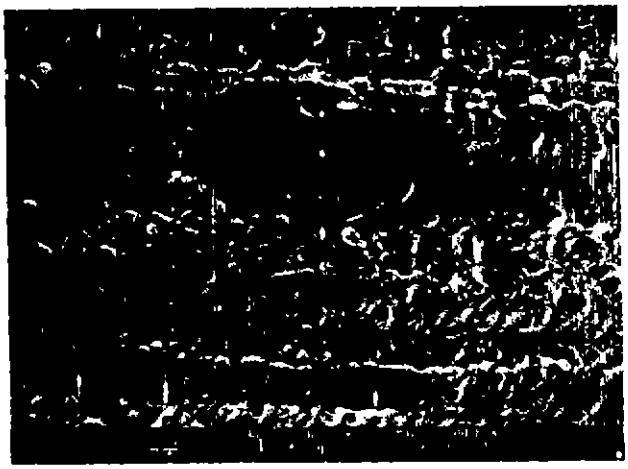
❑ Effect of melt index of PP

PP/SO 40 wt%, Coagulant : water at room temperature
Takeup speed : 53 m/min, Distance from spinneret to coagulation bath : 90 cm

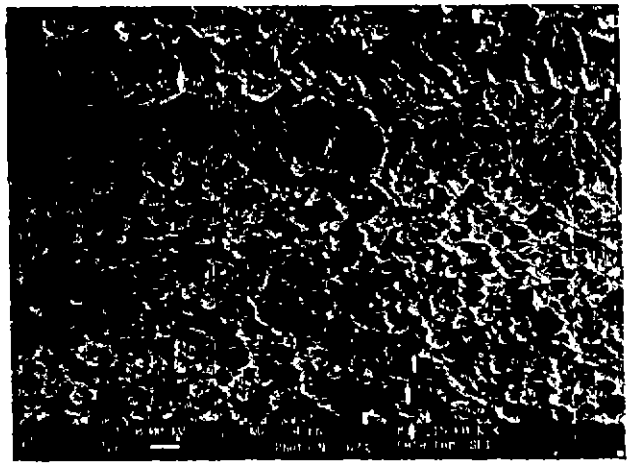
OUTER SURFACE



H730F(MI=8.4 g/10 min)

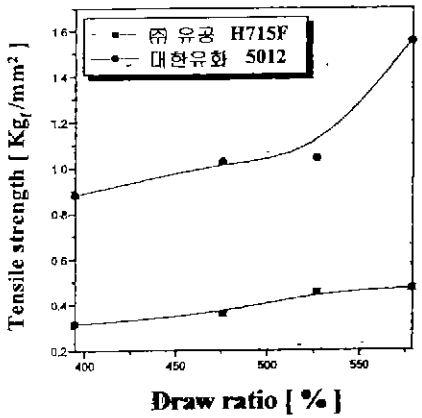


H235W (MI=5.7 g/10 min)



H715F(MI=2.0 g/10 min)

Tensile strength for each system



NA was added 1wt.% of PP

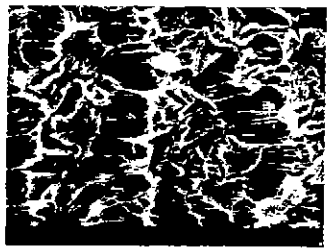
Heat treatment : 120 °C for 30min

Cooling at room temperature air

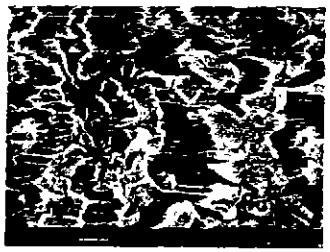
Polymeric Material Lab.

Structure variation depending on melt viscosity

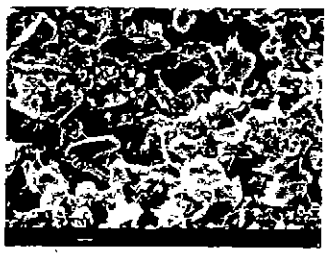
㈜ 유공 : H715F



Outer surface

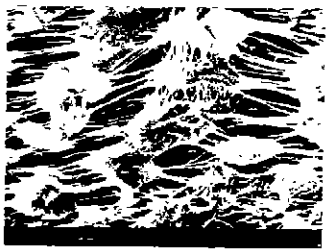


Inner surface

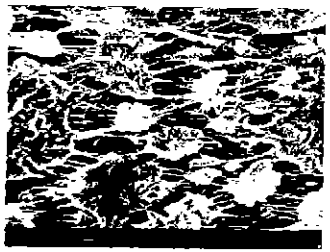


Cross section

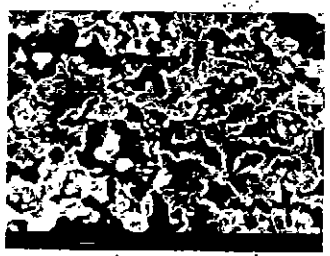
대한유화 : 5012



Outer surface



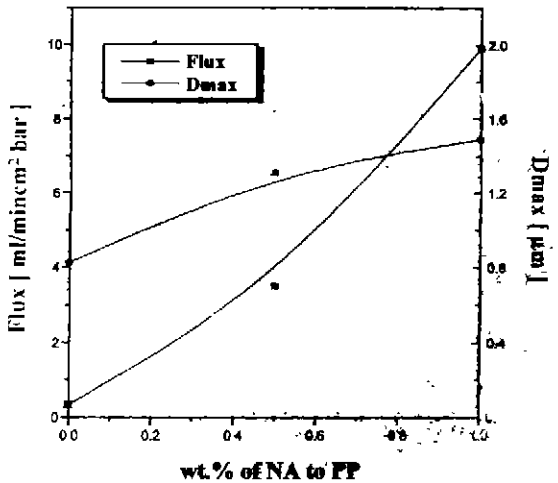
Inner surface



Cross section

Polymeric Material Lab.

Effect of nucleating agent on performance



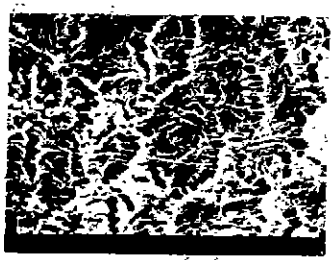
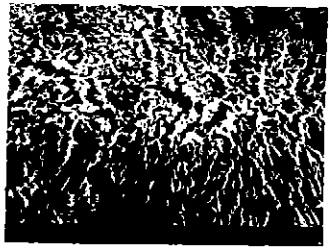
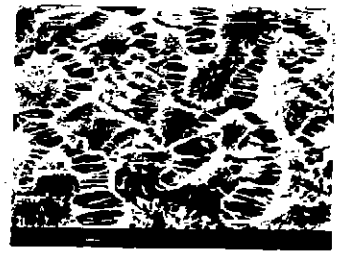
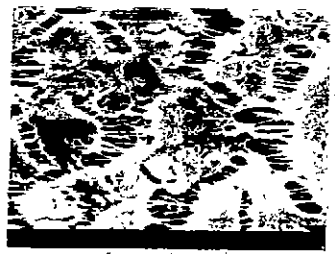
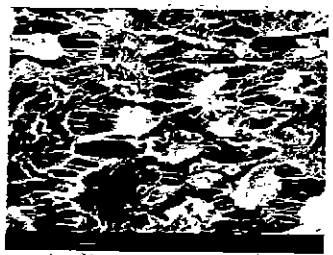
5012/SO 40/60

Heat treatment : 120 ℃ for 30min

Draw ratio : 579

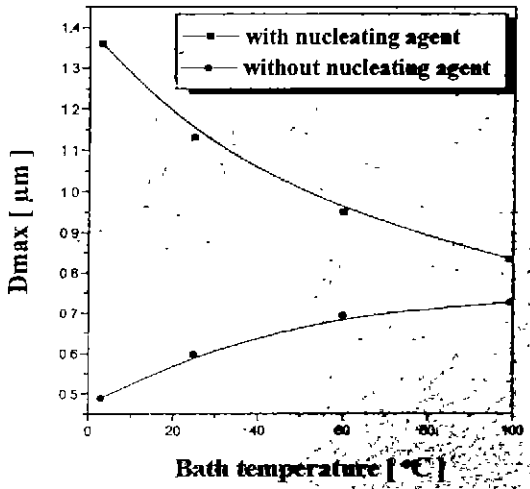
Polymeric Material Lab.

Structure variation depending on Nucleating agent

Outer Surface	Inner Surface
	
No use	No use
	
Nucleating agent 0.5%	Nucleating agent 0.5%
	
Nucleating agent 1%	Nucleating agent 1%

Polymeric Material Lab.

Effect of bath temperature on maximum pore size



5012/SO 40/60

NA was added 1wt.% of PP

Heat treatment : 120 ℃ for 30min

Coagulant : distilled water

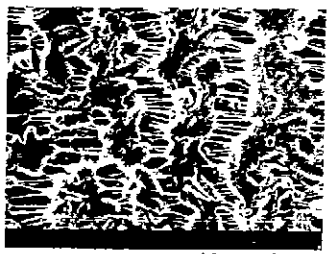
Draw ratio : 395

Polymeric Material Lab.

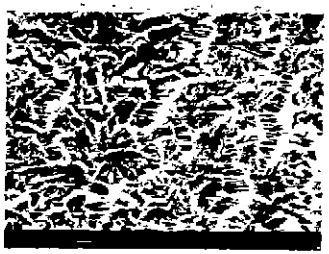
Structure variation depending on Bath temperature`

Outer Surface

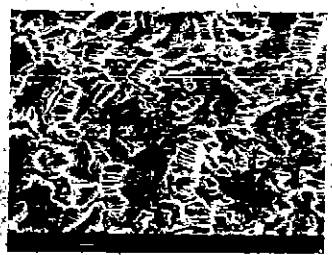
With nucleating agent



Bath temp : 3 ℃



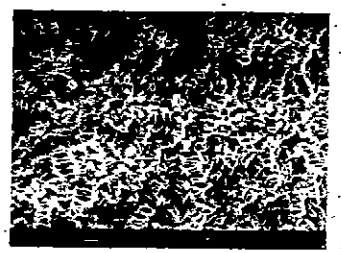
Bath temp : 60 ℃



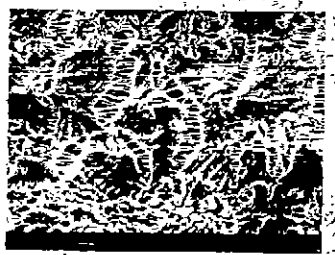
Bath temp : 100 ℃

Without nucleating agent

Bath temp : 3 ℃



Bath temp : 60 ℃

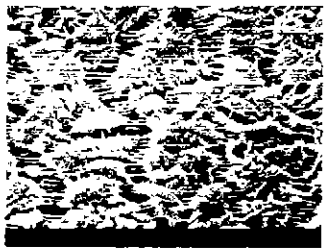


Bath temp : 100 ℃

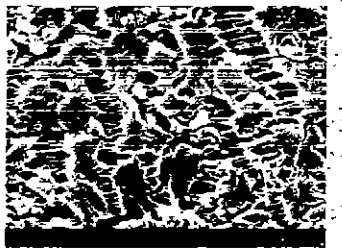
Polymeric Material Lab.

Structure variation depending on coagulant with soybean oil as a coagulant

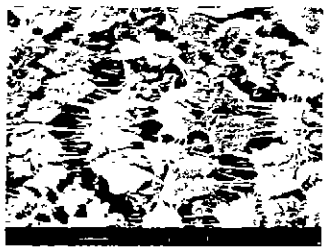
Outer surface



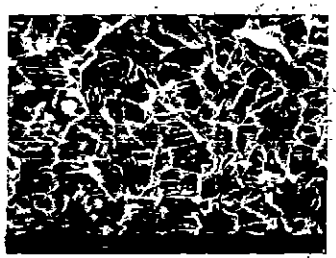
Draw ratio : 395



Draw ratio : 476



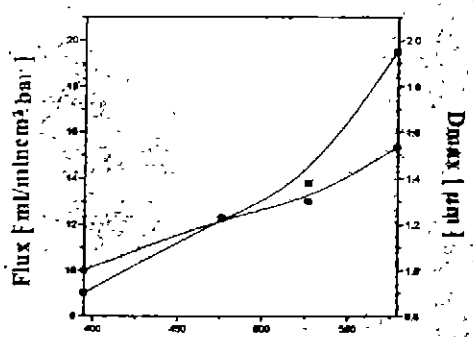
Draw ratio : 527



Draw ratio : 579

Polymeric Material Lab.

Effect of draw ratio on performance with soybean oil as a coagulant



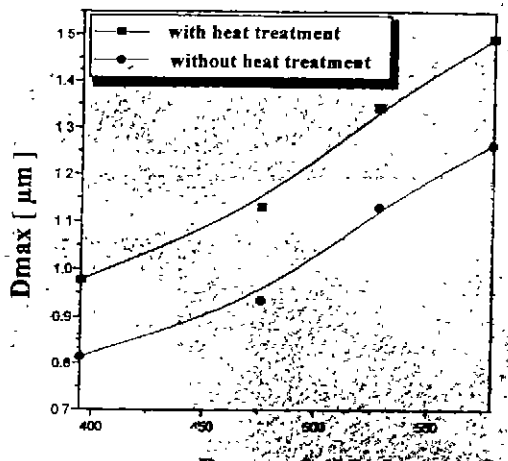
5012/SO 40/60

NA was added 1wt.% of PP

Heat treatment : 120 °C for 30min

Polymeric Material Lab.

Effect of heat treatment on maximum pore size



Composition : 5012/SO = 40/60

NA was added 1wt.% of PP

Cooling at room temperature air

Heat treatment : 120 °C for 30min

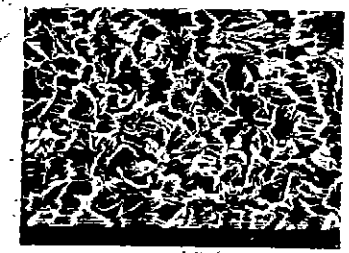
Polymeric Material Lab.

Structure variation depending on heat treatment

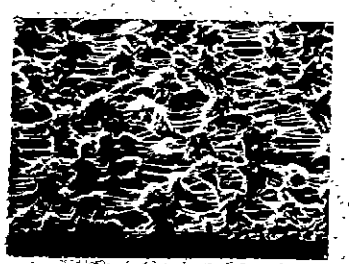
Outer Surface

Without heat treatment

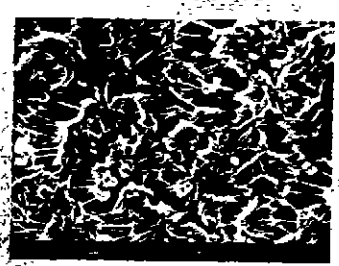
With heat treatment



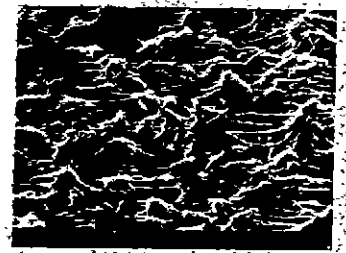
Draw ratio : 476



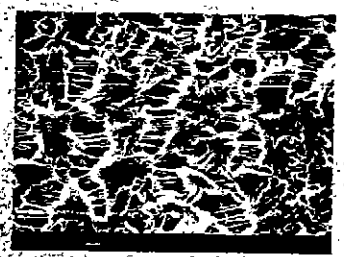
Draw ratio : 476



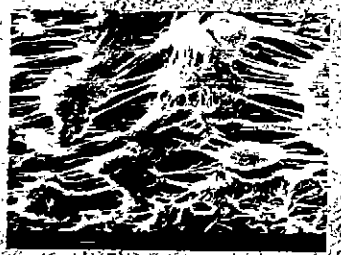
Draw ratio : 528



Draw ratio : 528



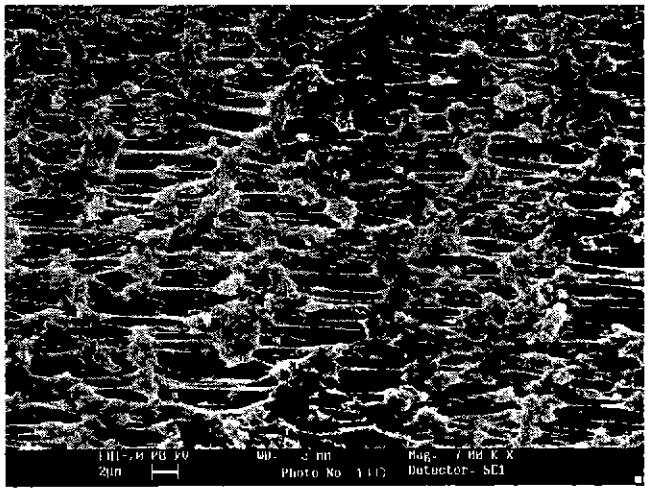
Draw ratio : 579



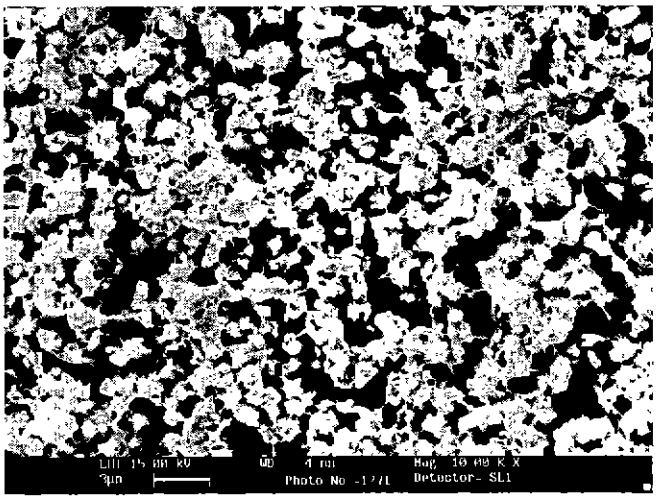
Draw ratio : 579

Polymeric Material Lab.

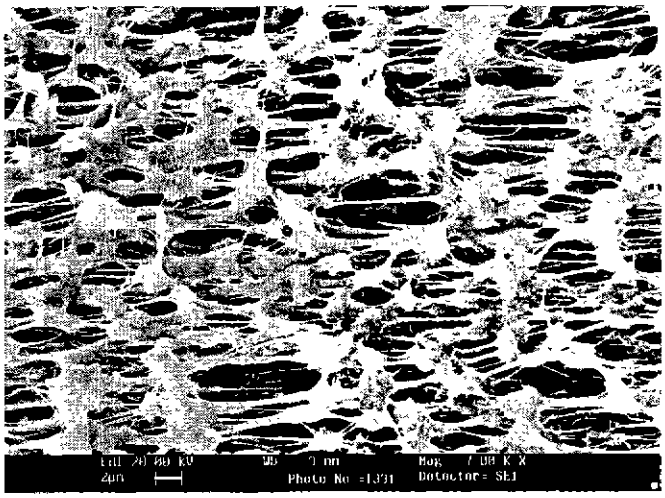
SEM Images of Optimized Membrane Produced by Korea Membrane Co.



inside



cross section



outside