Korea Institute of Science and Technology

Korea Institute Chemical Engineering Fall Meeting

Recycling study of the fiber stainless steel from grinding swarf by using supercritical fluids.

Jun Youl Yang[1], Chang Ha Lee[2], Youn Woo Lee[3], Ki Pung Yoo[4], Jong Sung Lim[4,*]

[1]Clean Technology Research Center, Korea Institute Science and Technology
[2]Dept. of Chemical Engineering, Yonsei University
[3]School of Chemical Engineering, Seoul National University
[4]Dept. of Chemical and Bio-molecular Engineering, Sogang University
*Corresponding author: limjs@sogang.ac.kr

Introduction

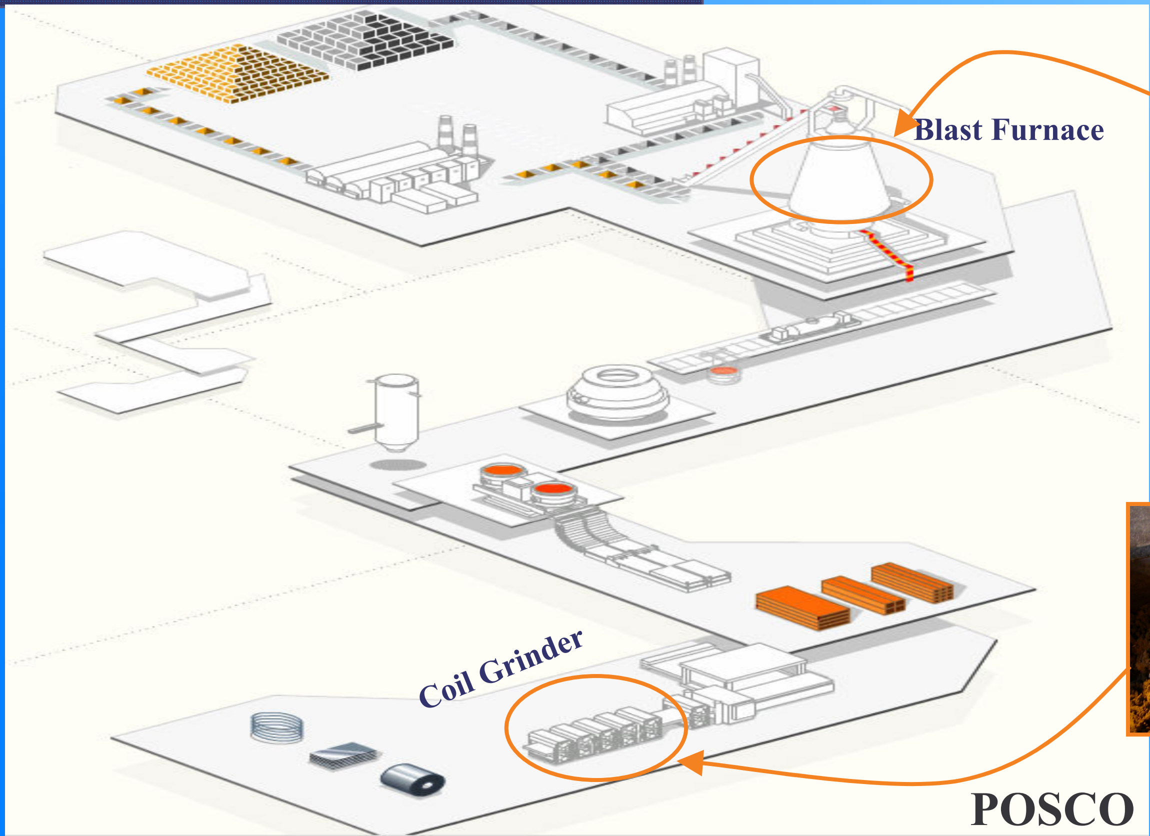
Grinding Swarf ?

The by-product produced from steel manufacturing process

Swarf +
Cutting oil

Specified Waste ! **Why?** **It's has cutting oil over 10% of total weight.**

Where was Swarf come from ?

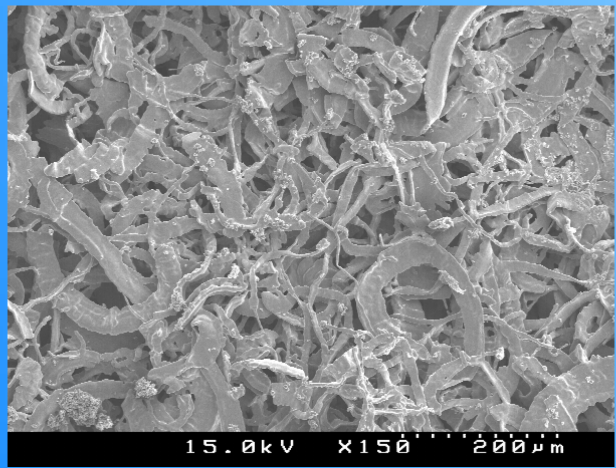


Briquette Swarf
: 670ton/year

Grinding Swarf
: 1250ton/year

Swarf Characteristics

√ **The mean size 150㎛ of the fiber structure**



SEM micrograph of Swarf

√ ***A fiber structure of Swarf is valued ten times more than same price metal materials because its manufacture is difficult and complex.***

Problems of the Current Disposal Method

- √ Low recovery rate for making steel ≈ 47 ~ 48%
- √ Produce of much fume
  because the swarf contains oil over 10wt% of total weight

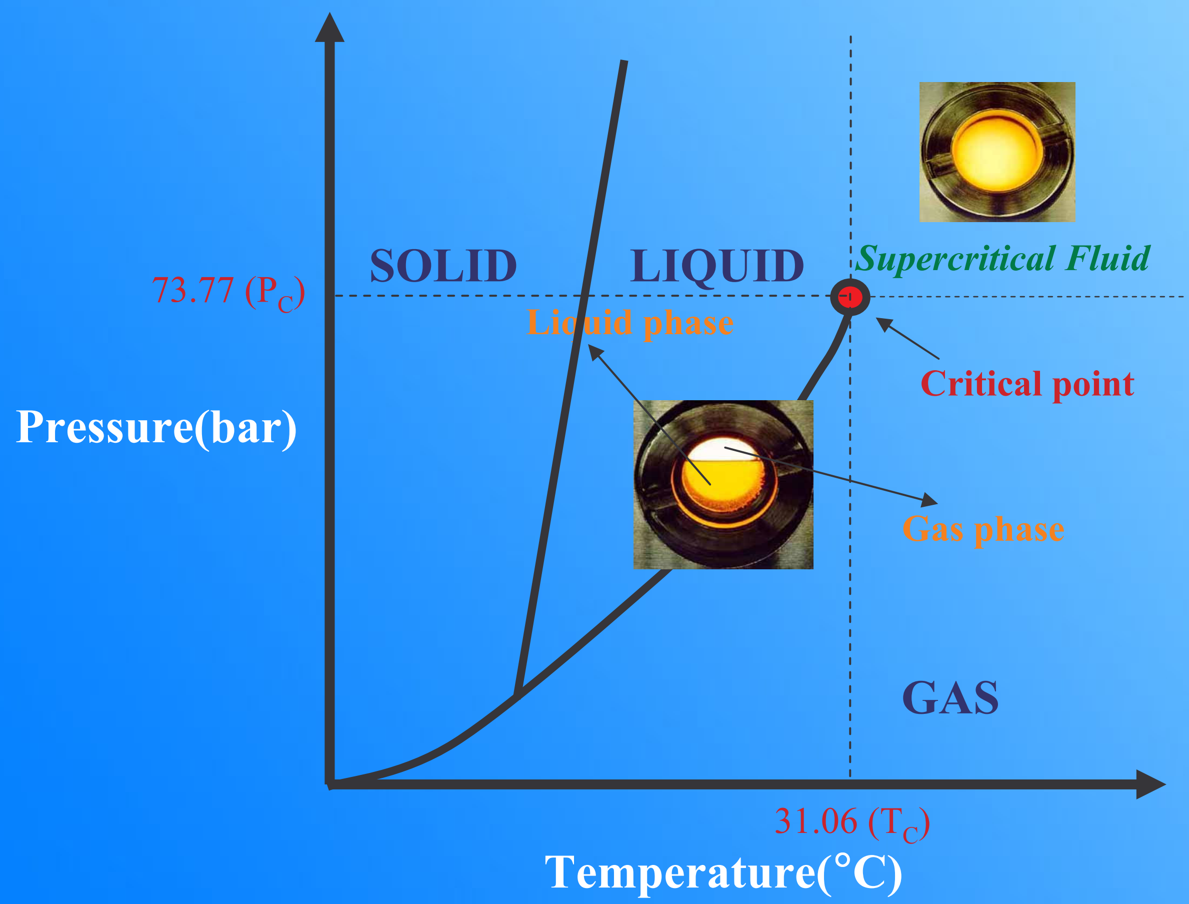
The Swarf Recycling Methods

- *Vacuum Thermal Extraction*
- *Solvent Extraction*
- *Supercritical Fluids Extraction*

Advantages of the Supercritical Fluids Extraction

- √ *No residual oil in grinding swarf*
- √ *Extracted oil can be reused without any refinement*
- √ *No deforming during extraction*
- √ *Environment-friendly and energy efficient*
- √ *Recycle of extraction fluid easily facilitated*
- √ *Adjustable solvent power*

Experiment



Good solvent power
No surface tension
Gas–like viscosity
Liquid like-density

Experiment Condition

Carbon Dioxide

√ Supercritical Condition
- T = 30 °C, P = 10, 15, 20, 25, 30MPa
- T = 40 °C, P = 10, 15, 20, 25, 30MPa
- T = 50 °C, P = 10, 15, 20, 25, 30MPa

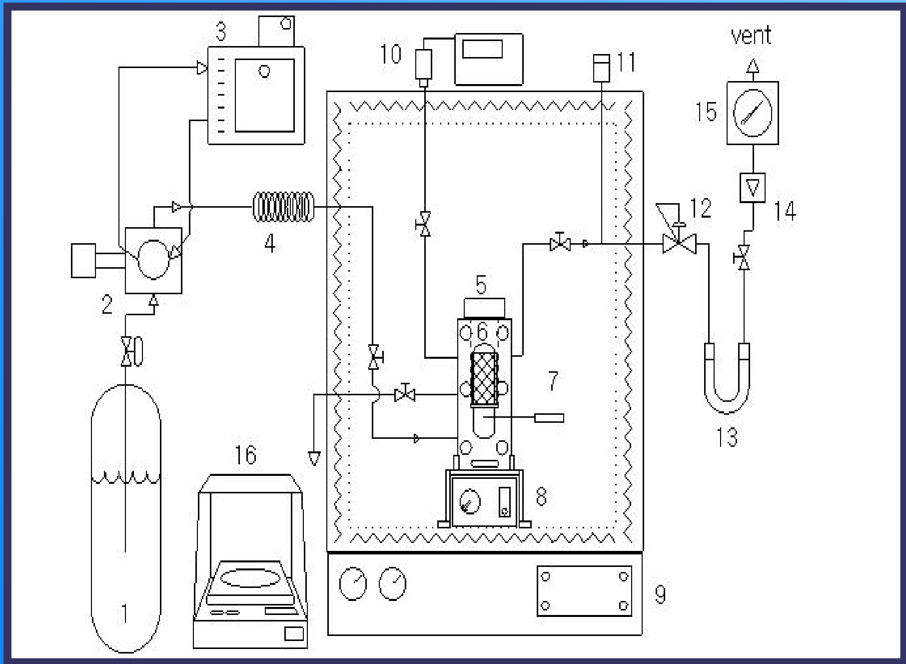
Propane

√ Subcritical Condition
- T = 80 °C, P = 5, 10MPa
- T = 90 °C, P = 5, 10MPa

√ Supercritical Condition
- T = 100 °C, P = 5, 10MPa
- T = 110 °C, P = 5, 10MPa

- Material : 13.4g swarf
- Solvent : carbon dioxide, propane

Experiment Apparatus



1. CO_2 or C_3H_8 cylinder
2. High pressure pump
3. Cooling circulator
4. Pre–heater
5. Extraction vessel
6. Metal basket
7. Thermocouple
8. Magnetic stirrer
9. Air bath
10. Pressure transducer
11. Rupture
12. Back-pressure regulator
13. Separator
14. Rotameter
15. Dry gas meter

Mathematical model

Single parameter model with linear desorption kinetics

Mass balance in bulk phase in the extraction cell

$$\frac{\partial C}{\partial t} + v\frac{\partial C}{\partial z} + \frac{1-\varepsilon}{\varepsilon}\rho\frac{\partial q}{\partial t} = 0$$

Linear desorption kinetics can be written as :

$$\frac{dq}{dt} = -k_d q$$

The mobile phase can be considered as an irreversible process Because of the lack of information on effective diffusivity of oil in $SCCO_2$ phase and the adsorption isotherms

The initial condition is :

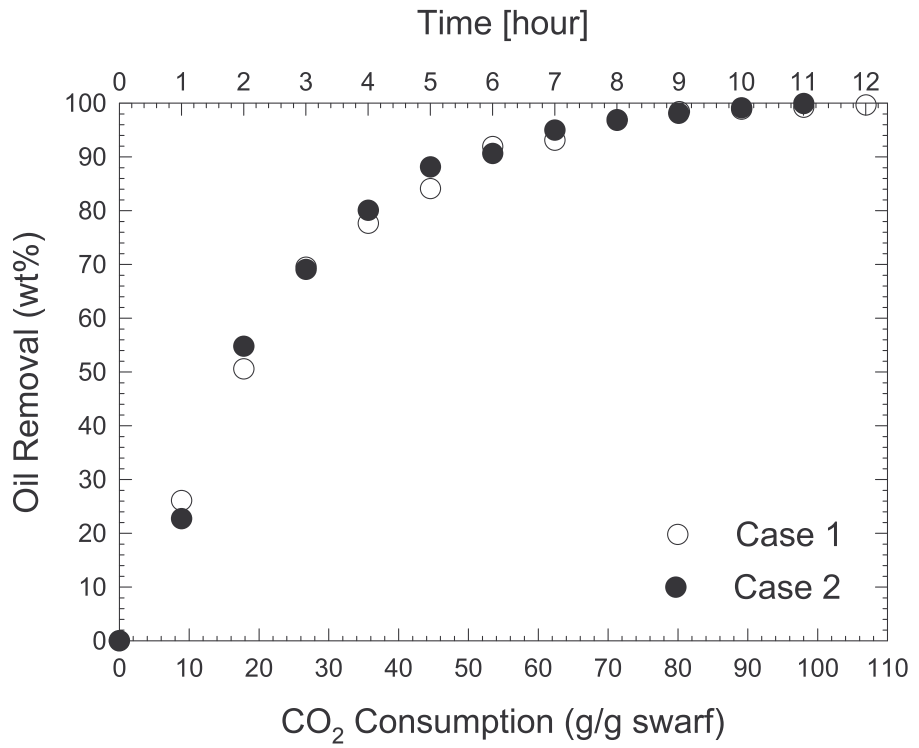
$$q = q_0 \quad at \quad t = 0$$

The desorption profile :

$$q(t) = q_o e^{-k_d t}$$

Result

The comparison of removal of oil in extraction vessel(case 2) with removal of oil from swarf(case 1) under 20MPa, 40℃.

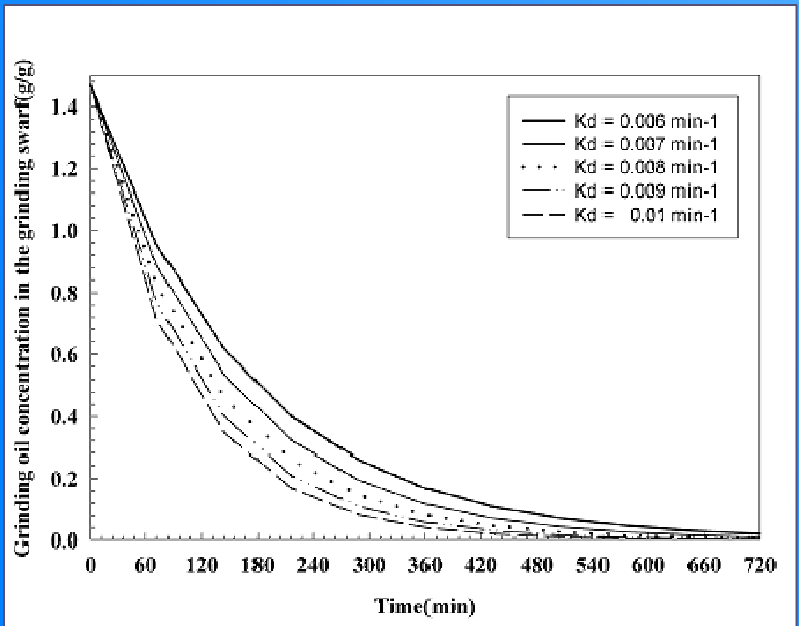


1. Carbon Dioxide

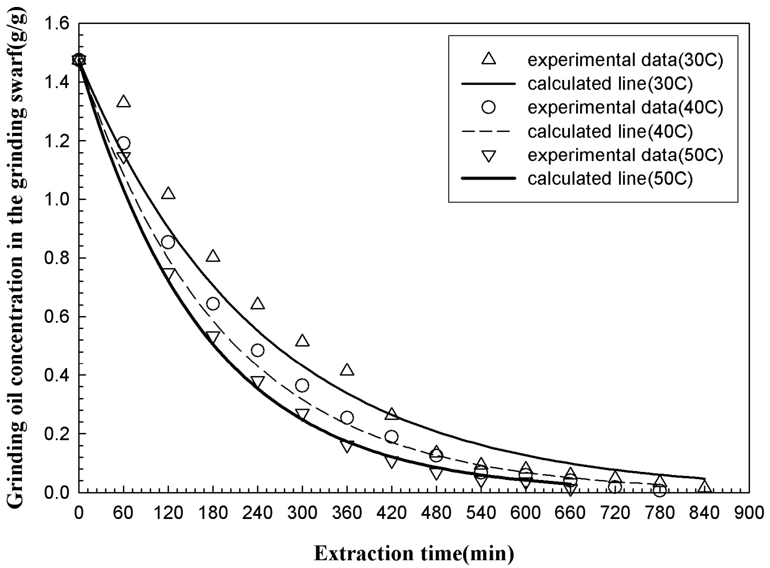
The parameters used in this model for supercritical CO_2 extraction

T(°C)	P(MPa)	q_0(g/g)	k_d(min-1)	t(min)	ρ_0(g/ml)
30	100	1.474	0.00409	840	0.772
	150	1.473	0.00593	720	0.847
	200	1.474	0.00633	720	0.891
	250	1.474	0.00659	600	0.923
	300	1.474	0.00728	600	0.949
40	100	1.474	0.00513	720	0.628
	150	1.473	0.00631	720	0.781
	200	1.475	0.00704	660	0.840
	250	1.475	0.00768	600	0.880
	300	1.475	0.00851	540	0.911
50	100	1.475	0.00594	660	0.386
	150	1.473	0.00678	600	0.701
	200	1.474	0.00785	540	0.785
	250	1.474	0.00896	480	0.835
	300	1.474	0.01037	480	0.871

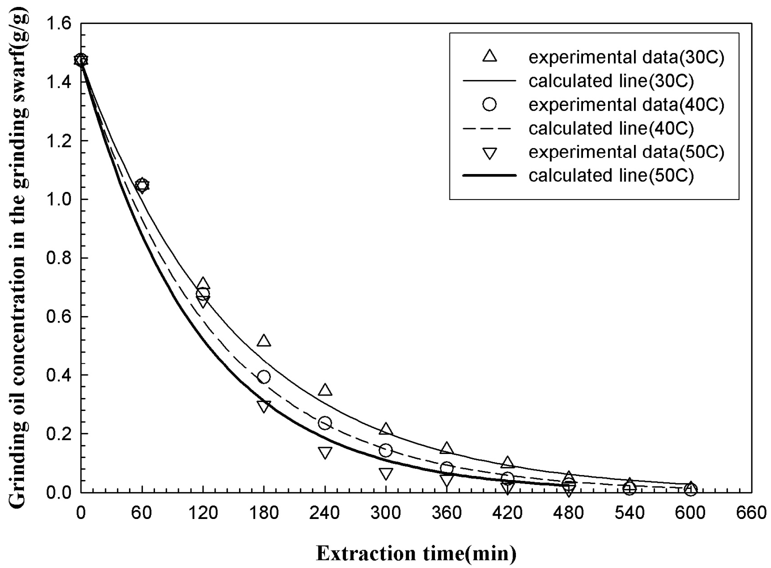
Grinding oil concentration at different k_d values



Effect of Temperature

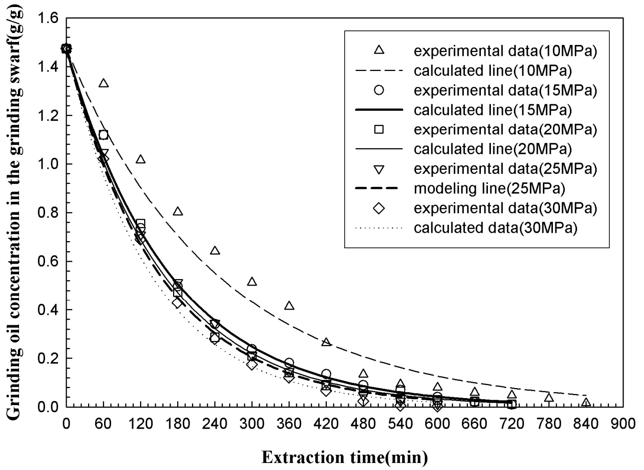


10MPa

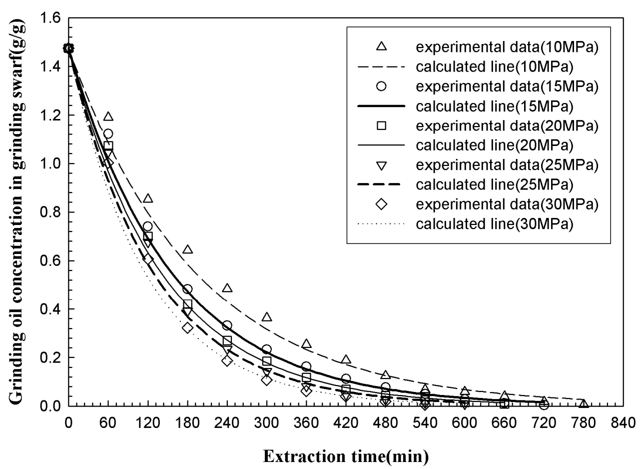


25MPa

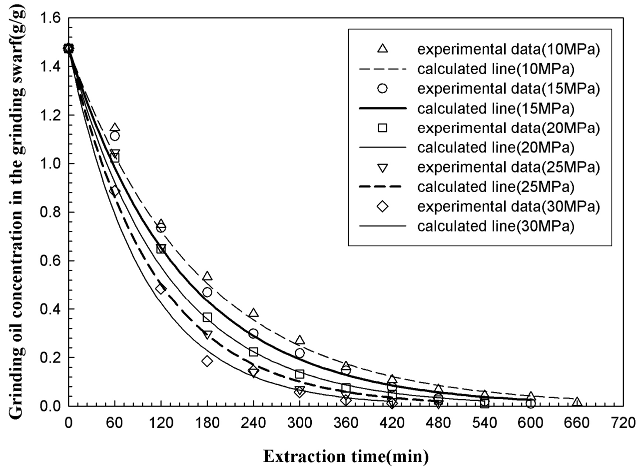
Effect of Pressure



30℃

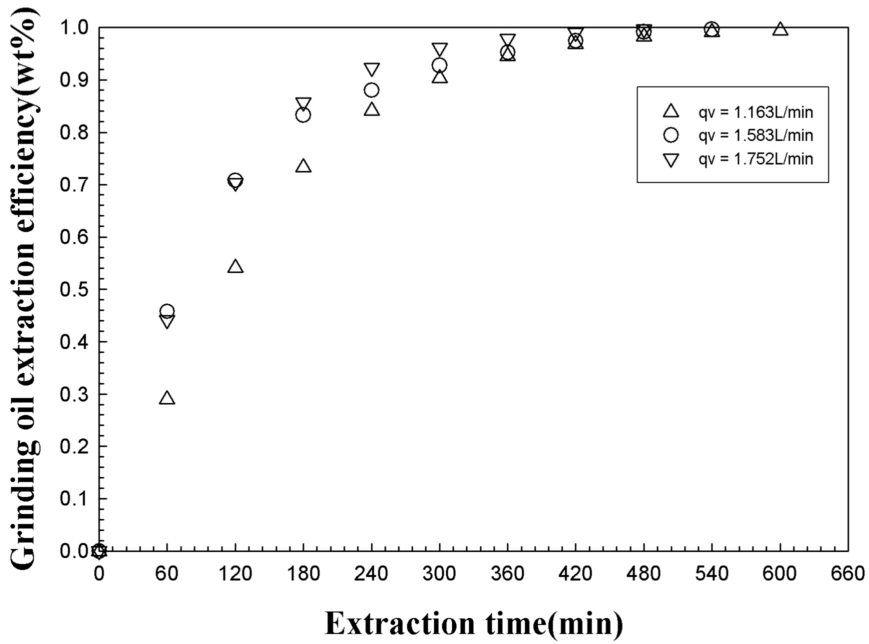


40℃



50℃

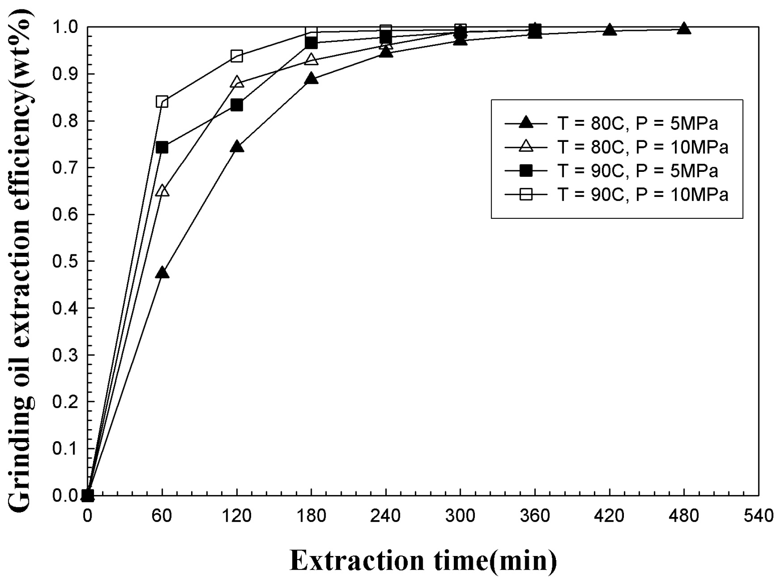
Effect of CO_2 flow rate



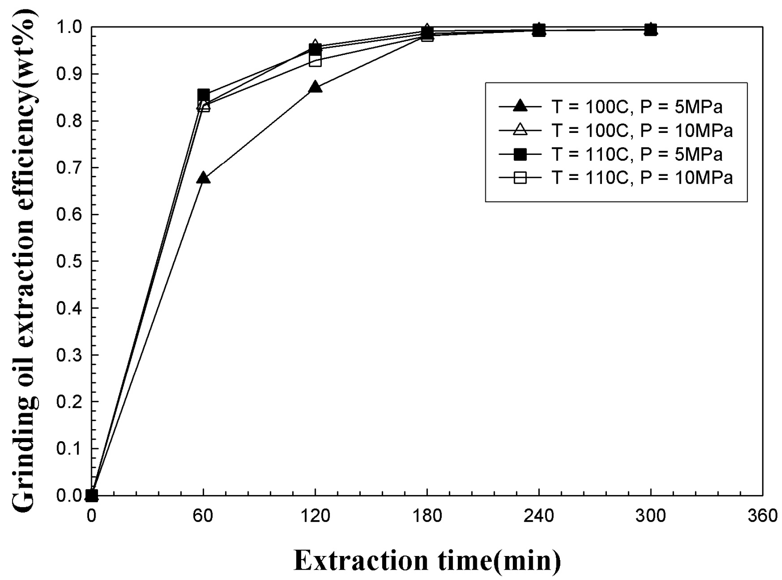
40℃, 20MPa

2. Propane

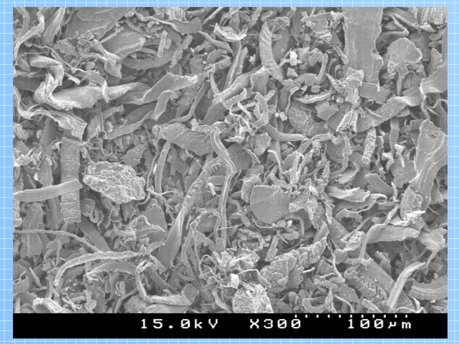
At Subcritical Propane state



At Supercritical Propane state

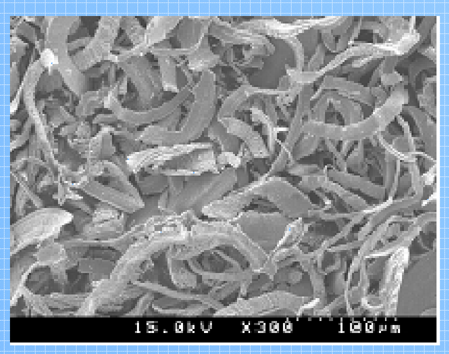


SEM Micrographs of the Swarf after extracting

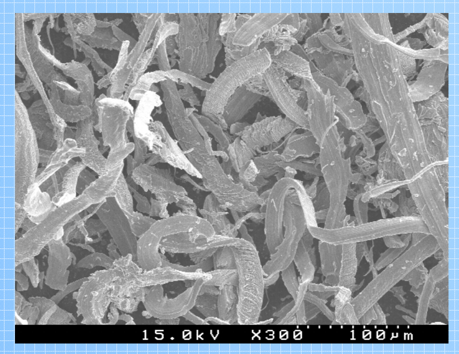


Vacuum Thermal Extraction

Solvent Extraction(n-Hexane)



Supercritical CO_2 Extraction



Subcritical C_3H_8 Extraction

Supercritical C_3H_8 Extraction

Conclusions

The feasibility of supercritical extraction to recycle the stainless steel by removing grinding oil from grinding swarf was tested.

In this work, the extraction efficiency depends on temperature and pressure. Also, as solvent flow rate increased, the extraction efficiency is increased. The experiment results was predicted by applying a one-parameter mathematical model assuming linear desorption kinetics. The predicted value showed good agreement with experimental data.

We could know from the SEM for the oil removed swarf that the steel fiber was deformed and oxidized by vacuum thermal extraction. However, by using supercritical fluids as a solvent, we could not only remove cutting oil from raw swarf, but also preserve the fiber's own characteristics of swarf effectively.

Alternative method : Supercritical Fluids Extraction