

## 세라믹사출 성형체로 부터 파라핀왁스의 분리

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**Separation of paraffin wax from ceramic injection molded part**Dong Hyun Kim, Youn Woo Lee<sup>1</sup>, Ki Pung Yoo, Jong Sung Lim\*

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## 1. Introduction

In ceramic injection molding process, Binder removal is one of the most critical steps in the CIM process due to defects that can be produced by inadequate binder removal, like bloating, blistering, surface cracking and large internal voids. [1] and the supercritical fluid has become an alternative for a conventional method. The disadvantages of conventional methods, such as thermal debinding or solvent extraction in ceramic injection molding process is that it require high temperature and long time so it is not energy efficient, deforming is happened and it is not environmentally acceptable [2], whereas the advantages of new method, supercritical debinding in ceramic injection molding, is that it is not toxic and recyclable and that it has simple process, low operating cost, short debinding time and low operation temperature. Extraction of organic binders (paraffin wax) from ceramic injection molded part by using supercritical fluids has proven to be very efficient. For this reason, recently, new method of extraction such as supercritical fluids extraction has been tried to remove binder from ceramic parts. Carbon dioxide has so far been the most widely used, because of its convenient critical temperature, cheapness, chemical stability, non-flammability, stability in radioactive applications, and non-toxicity. [3]

## 2. Theory

The diffusivity of a soluble wax through a porous injection molded part is evaluated by the Fick's diffusion-based model. We assumed that injection molded part is slab form with thickness  $l$ . Also, under the assumption that diffusivity of solute is constant and diffusing solute comes out through the slab faces and a negligible amount through the edges. The diffusion of a solute can be described according to Shewmon [4] by Eq. (1)

$$\frac{\bar{c}}{c_0} = \frac{8}{\pi^2} \exp\left(\frac{-D\pi^2 t}{l^2}\right) \quad (5)$$

$$\ln\left(\frac{\bar{c}}{c_0}\right) = \ln\left(\frac{8}{\pi^2}\right) - \left(\frac{D\pi^2}{l^2}\right) t \quad (6)$$

This expression can be used to determine  $D$  values. Table 1 shows the diffusivities determined in this work at supercritical  $\text{CO}_2$  conditions. From the table1, diffusivity increased with an increase in pressure at constant temperature.

### 3. Experimental method

The extraction vessel was made of 316stainless steel and had an internal volume of approximately 350  $\text{cm}^3$ . In the middle part of the cell, Pyrex glass windows of 18mm thickness are placed to allow the visual observation of the debinding procedure. A duplex high-pressure pump was used to feed  $\text{CO}_2$  continuously from a liquid  $\text{CO}_2$  cylinder to the extraction vessel in which the ceramic sample (green part) is loaded on basket. A pressure transducer and digital indicator measured the pressure in the cell. The system pressure was regulated within  $\pm 0.01$  MPa by using a back-pressure regulator which is located after the extraction vessel. There were two injection molded ceramics which are subtle different percentage of component. The first sample used in these binder removal experiments was about 1.4g and the second sample was about 1.35g. The green part was composed of alumina powder; its particle size was 400 nm, and binder materials. At the beginning, the flow rate of  $\text{CO}_2$  gas during the experiments was fixed to 1L/min for runs, and then the experiments were performed at different flow rate. The amount of binder removed was determined by measuring the weight of the debinded sample with a precision balance.

### 4. Results and discussions

The objective of this study was to suggest methods to improve supercritical fluids debinding. For this reason, removal rates were investigated at the different temperatures, pressure and flow rates. The flow rate was measured when the fluids went out from our process. So the flow rate was gas base.

Figure 2 shows that pressure effect at the constant temperature and flow rate. It is found that the debinding time of binder is increasing with increasing pressure at constant temperature and at constant flow rate because an increase in pressure while holding temperature result in enhancing the density of supercritical  $\text{CO}_2$ , so the solubility of wax increase.

Figure 1 shows temperature effect at constant pressure and flow rate. The result is caused that the supercritical fluids debinding should be performed at the temperature higher than the melting point of used wax (paraffin wax of the melting point about 329.15K). It is because above the melting temperature, the volatility of the paraffin wax becomes increased and the paraffin wax is easier soluble

to supercritical CO<sub>2</sub>. At figure 2, the line of 348.15K is closer to 338.15K than 328.15K. It shows that extraction rate does not increase as the debinding temperature increased above certain temperature or increase subtle amount as the debinding temperature increased. It was found that binder removal rate was affected by increasing density of the supercritical CO<sub>2</sub> rather than increasing volatility of the wax. From the results, supercritical debinding does not need high temperature, so energy can be saved during operating debinding process.

Figure 3 show effect of flow rates at constant temperature and pressure. As the flow rate is increasing up to 1L/min, the total extraction time is decreasing. However, when the flow rate is above 1L/min, the total extraction time is the same as the flow rate is 1L/min. From this result, it is found that 1L/min is adequate for binder removal from one ceramic injection molded part for our system.

Figure 4 is the result of debinding rate by different number of ceramic injection molded part. Temperature and pressure is constant. From the result, it is found that as the number of ceramic injection molded parts is increasing, extraction time of binder is multiplying. Total debinding time of two ceramic injection molded parts is two times longer than total debinding time of one ceramic injection molded part, and total debinding time of three ceramic injection molded parts is approximately three times longer than total debinding time of one ceramic injection molded part.

Table 1. Diffusivities of Paraffin Wax at the 20min of debinding time

Pressure(MPa)	Temperature(K)	Binder removal for 20 min(wt%)	Diffusivity
25	328.15	89.58	$1.73211 \times 10^{-10}$
25	338.15	95.53	$2.44671 \times 10^{-10}$
25	348.15	96.25	$2.595 \times 10^{-10}$
20	348.15	81.32	$1.23924 \times 10^{-10}$
15	348.15	33.26	$1.64096 \times 10^{-11}$

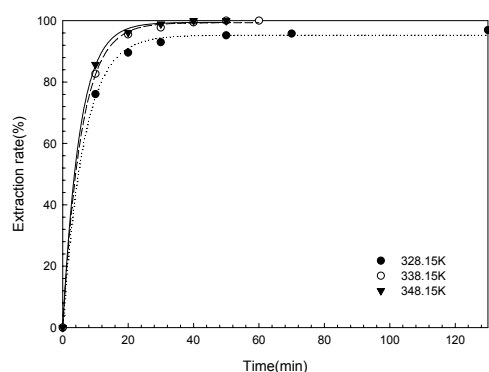


Figure 1. Temperature effect at 25MPa, 1L/min

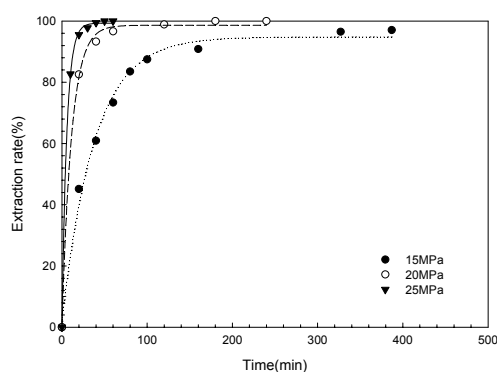


Figure 2. Pressure effect at 348.15K, 1L/min

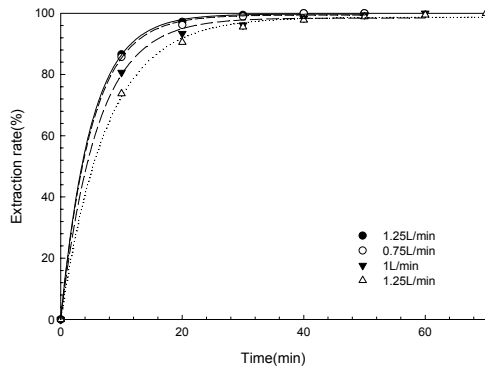


Figure 3. Flow effect at 348.15K and 25MPa

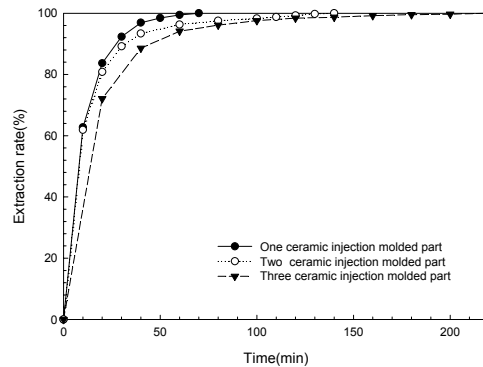


Figure 4. Effect of the number of ceramic

## 5. Conclusion

The purpose of our experiment was to find better debinding method and suggest alternative conventional methods by supercritical fluid. As the pressure increased, the extraction time decreased and the extraction efficiency become better. Diffusivity increased with an increase in pressure at constant temperature. And to make effective debinding, the debinding temperature should be above the melting temperature of paraffin wax. It was found that CO<sub>2</sub> of 1 L/min flow rate is adequate for one ceramic injection molded part. After removing binder from the ceramic injection molded part by supercritical CO<sub>2</sub>, the ceramic molded part showed no changes in the shape and the structure. Due to the only use of pure CO<sub>2</sub>, this debinding process is definitely environment-friendly operation. In one hour, all of the paraffin wax can be removed and the temperature of dinding should be higher than melting point. Therefore, the supercritical debinding is energy effective method and can beconsidered as an alternative debinding technology for a conventional method.

## References

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