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# EFFECT OF SOLVENT DEBINDING VARIABLES ON GREEN COMPACT WITH DIFFERENT BINDER FORMULATION

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## ABSTRACT

Effect of solvent debinding variables for extracting Fats, Oils and Grease (FOG) derivatives from different binder formulation feedstock were studied. The green compact with different binder formulation will be tested with two different organic solvents which are n-hexane and n-heptane at fixed solvent to weight ratio of 14:1 with temperature of 40, 50 and 60 °C for 10 hours. Effect of extraction temperature, solvents and time on green compact with different binder formulation will be monitored base on diffusion coefficient and weight loss. It is found that types of solvent, temperature and time has influence on extracting FOG derivatives from green compact with different binder formulation.

**Keywords:** fats, oils and grease derivatives, metal injection molding, solvent debinding, and diffusion coefficient.

## INTRODUCTION

Metal injection molding is a manufacturing process with a capability of producing complex geometry and intricate parts from mixing of metals or ceramic powder with combination of polymers, wax and surfactant(Kong et. al. 2012) with a few shot as compare to other fabrication process(Abolhasani & Muhamad, 2010; Afian et. al. 2012; Ahn, et. al. 2009). Due to its versatility, near net shape and less materials waste, it's becomes attraction to most researchers in exploiting it into new dimensions whether in terms of its binder, powder characteristic, injection molding conditions, debinding and sintering(Amin et. al. 2014). In metal injection molding (MIM) or powder injection molding (PIM), several steps need to be done, first is the mixing process where here the metal powder will be mixed with binder formulation with typical binder volume percentage lies between 20 to 50%(German & Bose, 1997) to form homogeneous feedstock. Secondly the produced feedstock will be injected to form desire shape and attain certain strength (German & Bose, 1997) before moving to the next phase. Debinding process is the third process where contributes in removing the binder from the components following by the sintering process.

Organic binder is present to facilitate flowability of the powder-binder suspension into mould cavity during injection molding and to keep the particles attached inflexibly. The most widely used methods in removing this binder are by thermolysis(Liu & Tseng, 1998), catalyst(Weil, et. al. 2006), wicking(Enneti, et. al. 2012) and solvents(Zaky, 2004).The removal of organic binder usually termed as debinding process is known to be critical in obtaining better shape retention, no defects and desired microstructure(Liu and Tseng 1998). Solvents extraction of one components of binder has been widely used to create porosity in green bodies for easily removing the backbone binder during thermal debinding.

Types of solvent used also influenced the integrity of the green compact since some types of solvent has strong effect on the backbone binder (polymers) which results in cracking and distortion of green compact. Therefore, the thermal removal of insoluble binder components will be finished in a much shorter time without endangered the integrity of the green compact. Consequently the combination of solvent debinding and thermal debinding can be time saving (Tsai & Chen, 1995).

Thus, the objective of the present study is to extract the FOG derivatives from green compact in the shortest time without scarifying the quality of the brown compact. Also in order to compare the effect of binder formulation on the shortest time of FOG removal from green compact, two feedstock with different binder formulation were injected to form green compact and being subjected with the same solvent debinding variables.

## EXPERIMENTAL

### Powder and Binder Characteristic

Water atomized stainless steel powder (D50=6µm) was chosen since the particle size distribution (Table-1) is between 2µm and 11µm which is suitable for metal injection molding (German & Bose, 1997). Powder morphology used in the present work is shown in Figure-2. The particles have irregular in shape hence will gives better shape retention during binder removal but lower packing density (Aggarwal, et. al. 2006). Mixing was done using Brabender Plastograph EC mixer at temperature of 175 °C at 30rpm for 90min. Mixing temperature must be at the lowest degradation temperature between binders components and higher than the highest melting components of the binder components.

**Table-1.** Water atomised stainless steel (316L) powder characteristics.

| Powder size and density | | | | | |
|---|---|---|---|---|---|
| Powder | Size | $D_{10}$ | $D_{50}$ | $D_{90}$ | Density |
| SS316L Epson Atmix Corp | 6.0 μm | 2.87 μm | 5.96 μm | 10.65 μm | 8.0471 g/cm³ |
| Powder chemical composition (% wt) | | | | | | | |

| Ci | Si | Mn | P | S | Ni | Cr | Mo | Cu |
|---|---|---|---|---|---|---|---|---|
| 0.0 27 | 0. 84 | 0. 19 | 0.01 6 | 0.01 2 | 12.2 0 | 16.40 | 2. 10 | 0. 03 |

**Table-2.** Characteristics of the binder system.

| Binder | Density (gcm⁻³) | Melting Temperature (°C) | Degradation Range (°C) |
|---|---|---|---|
| Polypropylene (PP) | 0.90 | 140 | 350~470 |
| Sewage Fat (SF) | 0.90 | 50 | 270~360 |

**Table-3.** Volumetric powder loading 60% and proposed binder formulation for feedstock.

| Formulation | Compositions and contents | |
|---|---|---|
| | Powder (Volume percentage) | Binder ratio (Volume percentage) |
| F1 | 316L Stainless Steel (60%) | PP (60) + SF (40) |
| F2 | 316L Stainless Steel (60%) | PP (20) + SF (20) |

Binder system characteristic is shown in Table-2 with binder formulation shown in Table-3. This two binder formulation will be mixed with 60% volumetric powder loading of stainless steel SS316L powder which is approximately 5% (German & Bose, 1997) below the critical powder volume concentration(CPVC) of water atomised SS316L powder (CPVC = 64.8%)(Ibrahim, et. al. 2011) to form a feedstock and being injected into green compacts shown in Figure-2(a).
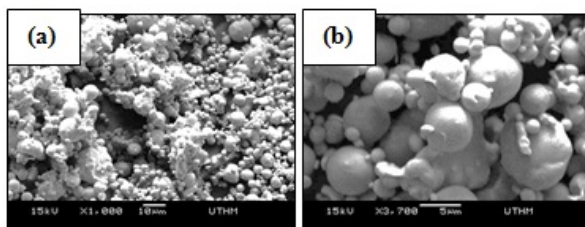


**Figure-1.** (a) SEM micrograph of SS316L (PF-10F) at 1000x (b) SEM micrograph of SS316L (PF-10F) at 3750x.
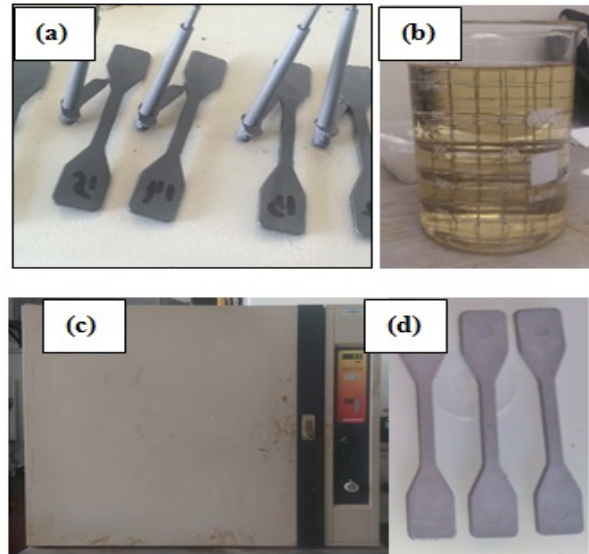


**Figure-2.** Test specimens (a) Part after injected (b) Part being submerge into solvent  (c) solvent with green compact being heated in the oven (d) Green compact after solvent.

Solvent debinding of the green compacts was done by submerging the components into the solvents and heated inside the oven shown in Figure. 3(b). Weight loss of the green compact is then being measure every 1, 3, 6 and 10 hours by the methods of replication.

**RESULTS AND DISCUSSION**

**Effect of Temperature on Weight Loss and Diffusion Coefficient**

Temperature effect is one of the important variables in the solvent extraction process. Figure-3, 4, 5 and 6 show the effect of the extraction temperature on the percentage weight loss and the diffusion coefficient of FOG derivatives by using different organic solvent at a fixed solvent to feed ratio (S/F=14:1) for 1 hour. Diffusion coefficient against time being plotted base on Eqn. 1.

$$\frac{C_a - C_o}{C_i - C_o} = \frac{8}{\pi^2} e^{\frac{-\pi^2}{h^2}Dt}$$

(1)

Where $C_a$ is the average concentration of binder remaining on compressed part, $C_i$ is the initial concentration of binder, $C_o$ is the boundary condition (zero), $t$ is the debinding time, $D$ is the debinding coefficient and $h$ is the thickness of the compressed part (Nanjo et. al., 1993 cited in Zaky, et. al. 2009) which was taken from Fick's 2nd Law. Fick's 2nd Law stated that fluid migration in the matrix occurs mainly through diffusion and concentration drive this migration(Liu, et. al. 2015).

From the weight loss against temperature profile (Figure-3 and 4), as the extraction temperature increases,

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the percentage weight loss and diffusion coefficient (Figure-5 and 6) also increases. This is due to the highest penetration rate of solvents which results from higher kinetics energy of solvent molecules. Thus at higher temperature more microscopic cracking network and pore size distributions were created in F2 since it has higher volumetric percentage of FOG compare to F1 (Caré, 2008).
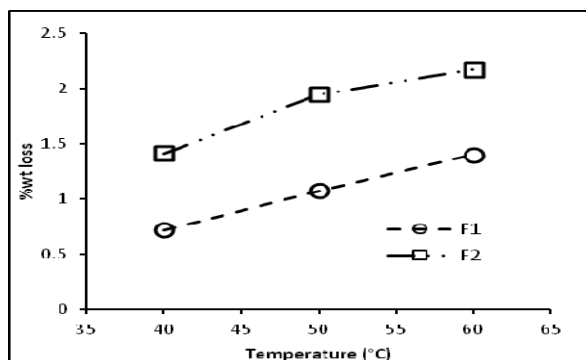


**Figure-3.** % weight loss of green compact against temperature using Hexanes solvent for different binder formulation during 1st hour.
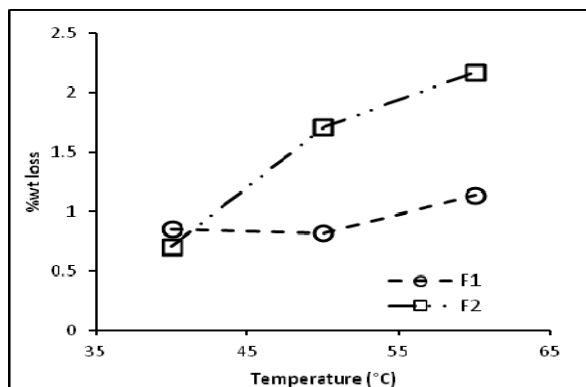


**Figure-4.** % weight loss of green compact against temperature using Heptanes solvent for different binder formulation during 1st hour.
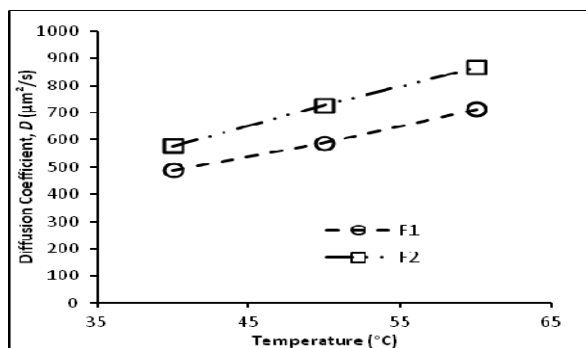


**Figure-5.** Diffusion coefficient of FOG derivatives against temperature using Hexanes solvent for different binder formulation.
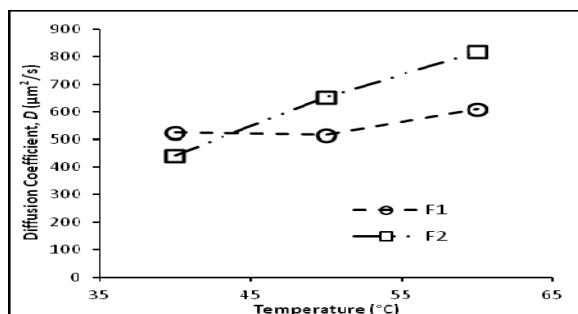


**Figure-6.** Diffusion coefficient of FOG DERIVATIVES against temperature using Heptanes solvent for different binder formulation.

**Effect of Solvents on Weight Loss and Diffusion Coefficient**

From Figure-7, graph profile of F1 solvent debound using both heptane and hexane was plotted and it can be seen that hexane solvents gives the highest weight loss as compare to heptane. This is also true for F2 binder formulation where hexane shows slightly higher in weight loss as compare to heptane between 1 to 3 hours period. This indicates that hexane solvents has better extraction rate of FOG as compare to heptanes and this might due to lower carbon number of hexane which increase the solvents diffusion rate into the FOG (Zaky, 2004). From the Figure-8 of diffusion coefficient also shows that hexane gives higher diffusion coefficient as compare to heptane for both binder formulation.

Although F2 binder formulation has the highest weight loss and diffusion coefficient, solvents type did not give any effect on binder formulation since percentage weight loss was depends on quantity of FOG inside the green compact. Higher diffusion coefficient also influenced much on concentration of FOG inside the green compact which drives its. Therefore it can be conclude that types of solvent did not gives any significant influence on extracting FOG from the green compact with different binder formulation. Hexane gives the highest diffusion coefficient and weight loss as compare to the Heptanes.

Effect of debinding time on Weight Loss and Diffusion Coefficient binder formulations

Duration of debinding time plays significant role since it can reduce the time consuming and increase the productivity of the process. From Figure-7, weight loss is proportional to time until at certain period of time. Both binder formulation shows the weight loss increment for the first 6 hours. F2 binder formulation weight loss stabilized after 3 hours debound as compare to F1 which took for about 6 hours to stabilized. This can be seen by calculated the difference of weight loss of both feedstock between 3 and 6 hours after solvent debound process. The difference of weight loss for F2 between 3 and 6 hour is about 0.023% as compare to F1 which is 0.174% (Figure-7). The lower the difference indicates that the extraction process become stabilize much faster as compare to the highest one.

The results of the faster extraction time has been compare with diffusion coefficient profile Figure-5 and Figure-8 where diffusion coefficient is higher during the first hour of debound process for F2. This was due to higher concentration of FOG initially and decreasing with time. Since concentration of FOG drives the migration, as the concentration of FOG decreasing the diffusion coefficient also become lower with time. Results of this quick migration influence the pores size and quantity developed inside the green compact which accelerates the debound proces(Liu & Tseng, 1998; Yang *et al*., 2006). Tortuosity might also influence the shortest debinding time of the FOG from F2 since porosity structure channel created is more homogeneity as compare to F1. This indicates that tortuosity increases with adding more polymer inside the green parts as in F1(Yumei Yong *et al*., 2013). Therefore F2 binder formulation gives much less time for solvent debound process for the same part shape and dimensions.
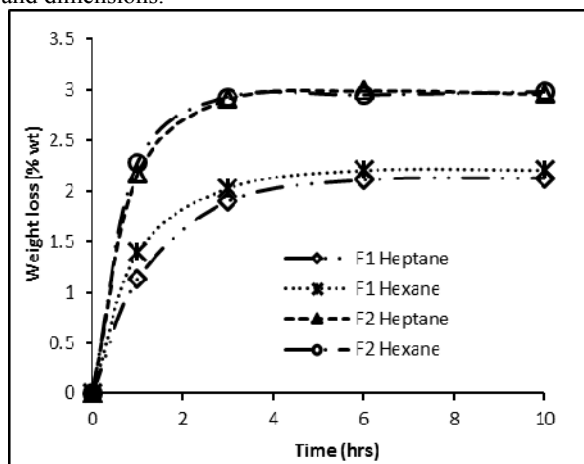


**Figure-9.** DSC analysis of F2 feedstock where FOG DERIVATIVES Tm=51.8 ℃ and Tg=35.7 ℃, PP Tm=151.3 ℃ and Tg=106.2 ℃.



**Figure-7.** % weight loss against time for different binder formulation and organic solvents at extracting temperature of 60 ℃.
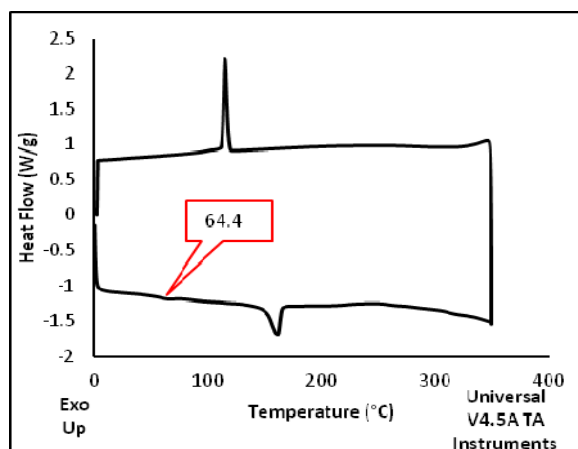


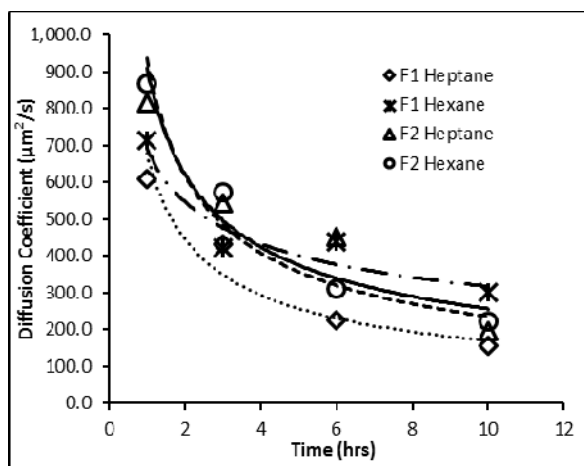**Figure-10.** DSC analysis of F2 feedstock after 1 hr solvent debound.



**Figure-8.** Diffusion coefficient against time for different binder formulation and organic solvents at extracting temperature of 60 ℃.



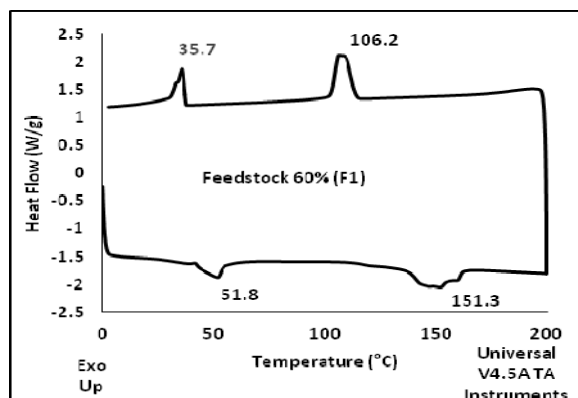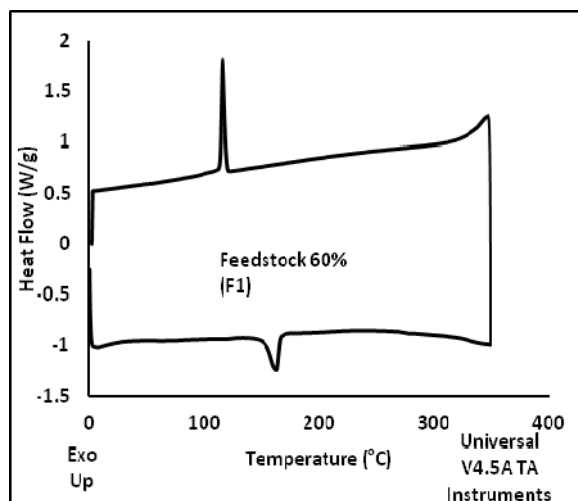**Figure-11.** DSC analysis of F2 feedstock after 10 hrs solvent debound.

**Differential Scanning Calorimeter (DSC) of Solvent Debinding**

Further analysis of the FOG DERIVATIVES loss during solvent debound can be monitored using DSC where can be determined by the peak of the melting point of the binder components. Figure-9 shows the F2 feedstock before solvents debound. Figure-10 and 11 shows the evolution of green compact after extraction for 1 and 10 hours. After 1 hour, the FOG derivatives has some FOG portion remaining inside the green compact and not fully removed by the hexanes and this can be observed by small secondary peak at the endothermic side. After 10 hours no secondary peak was observed at the endothermic side. this can be conclude that at durations of 3 to 10 hours, the FOG DERIVATIVES has been completely removed from the green compact by hexanes solvent at extraction temperature of 60ºC. Evolution of binder matrix can be observed by the Scanning Electron Microscopic (SEM) where small pores or channel can be monitored from Figure-12 to 14.
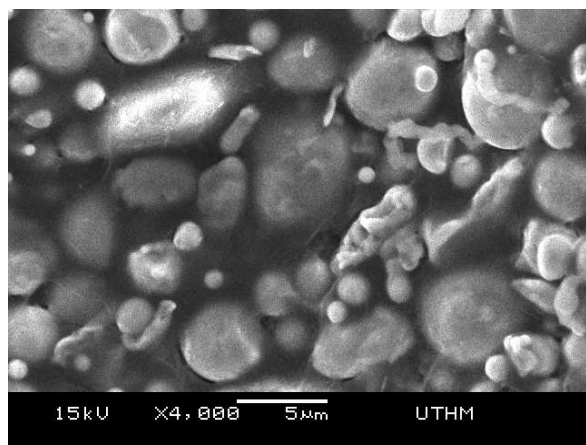


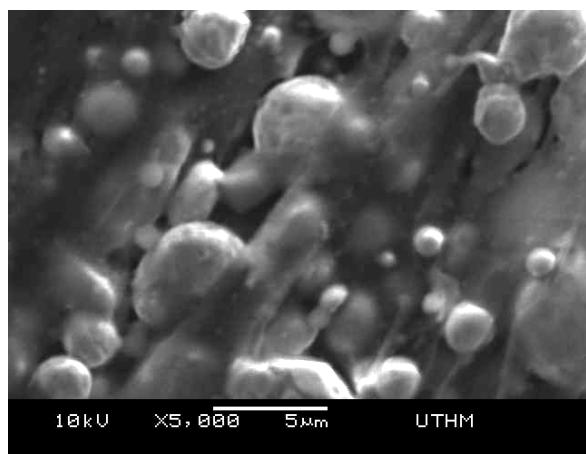**Figure-12.** Green compact of F2 feedstock morphology.



**Figure-13.** F2 feedstock morphology after 3 hrs solvent debound.
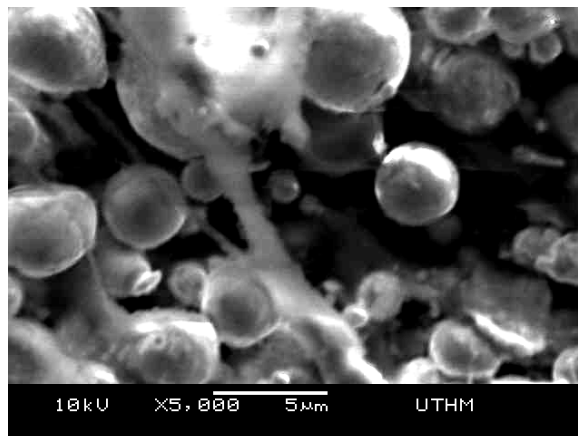


**Figure-14.** F2 feedstock morphology after 10 hrs solvent debound.

**CONCLUSIONS**

Green compact bodies consisting of PP and FOG derivatives as binder with volumetric powder loading of 60% were debound through solvent extraction with n-hexanes and n- heptanes. Binder formulation is important since it will enhance the diffusion coefficient which results in increasing the debinding rate. Selection of organic solvent of higher solubility such as n-hexane is also important to enhance the debinding rate. The extraction temperature of 60ºC, solvent to feed ratio of 14:1 by weight and extraction time of 3 to 10 hours are the most suitable conditions for extracting the FOG derivatives from the binder mixture without affecting on the shape maintenance of green compact bodies. Differential Scanning Calorimeter (DSC) showed also, all the FOG derivatves constituents are completely extracted from the binder mixture which confirmed the above findings. SEM observations show the creation of a large amount of pores after solvent debinding which facilitate the subsequent thermal debinding without endangering the integrity of green compact body.

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