Experimental Study of Solvent Debinding on Water Soluble PEG Behavior for Water Atomised SS 316L Compact

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Abstract—Growth of environmental friendly binders like Polyethylene glycol (PEG) has received considerable interest because it is a water-soluble binder which is safe to the environment. From the study, PEG removal in water during solvent debinding was observed as a function of temperature against time by determining the density value of compacts. SS 316L water atomised powder around 5 μm particle size with powder loading 61 vol. % were mixed using 73 vol. % of PEG, 25 vol. % of PMMA and 2 vol. % of SA. The injected compact using injection moulding machine was solvent debinding in water leaching at different times and temperatures. The effect of temperature and time on the leaching performance of PEG in water was investigated through relative density observations and microstructural analysis using an optical microscope (OM). The amount of PEG removed increased with time for all temperatures. Observations using different immersion temperatures: 40, 50, 60, 70 and 80 °C with different immersion times were used: 2, 4, 6, 8, and 12 hours (h) for removing the PEG binder. After 12 h, the brown compact at the highest immersion temperature of 80 ºC showed 100 % PEG loss. The microstructure showed that as the time of debinding increased, the shape of the pores and particles became clearer with the remaining binder component of PMMA polymer attached to the particles as most of the PEG was removed from the green compact. The relative density values of the brown compact were reduced when compared to the green compact which ascertained the removal of the PEG binder during the solvent debinding process.

Keywords— polyethene glycol (PEG); SS 316L water atomised powder; metal injection moulding; solvent debinding; density measurement; microstructural analysis.

1. INTRODUCTION

The MIM process usually occupies four steps which are feedstock mixing, injection moulding, debinding and sintering. The third process in MIM is a debinding process which functions to remove the polymer binder using solvent or heat [1, 2]. Debinding is a critical process to remove the binder from the green compact as the compact can have defects such as swells

and cracks if unsuitable debinding variable is conducted. The solvent extraction process is mainly used as it can save time and cost for solvent and thermal debinding processes. There are several causes that can control the time taken for the process of solvent extraction such as solvent temperature, type of solvent, flow rate of solvent, and sample thickness [3]. The binder system consists of PEG and PMMA which are chosen as they can be easily removed during solvent debinding and thermal debinding [4]. The PEG binder is known for being safe to the environment and acts as a water soluble binder [5, 6]. The PEG can help to decrease feedstock viscosity and increase replication capability [7]. The PMMA is used to maintain the shape of parts after injection moulding and debinding [8]. The SA acts as a lubricant as it is a surfactant which improves binding properties such as spreading, surface wetting, and binder strengthening [9]. Previous research conducted by Hwang et al. […] concluded that the thickness of a cross-section determined the debinding rate, while the size of a particle did not influence viscosity for spherical powders, and hence not the rate of debinding [10, 11]. Solvent debinding can be controlled by immersion temperature and immersion time [12]. This study aims to investigate the effect of temperature and time on the leaching performance of PEG in water. Relative density was determined from observations and microstructural analysis using an optical microscope (OM). Based on the results obtained, the immersion temperature and time of solvent debinding for the green compact based on the leaching performance of PEG in water are proposed.

2. METHODOLOGY

The material used in this research was SS 316L water atomised powder from Epson Atmix Corp. The shape of the particles is irregular with a mean particle size of 5 μm and tap density of 8.0471 g/cm^3. The powder loading for the feedstock was 61 vol. %. According to Ibrahim et al. [13, 14], the best powder loading for SS 316 L with 73 vol. % PEG, 25 vol. % PMMA, and 2 vol. % SA is between 59.8 – 62.8 vol.%. The binder composition of 73 vol. % PEG, 25 vol. % PMMA, and 2 vol. % SA was used as it has been effective in moulding a mixture of ceramic and metal powders as stated in [15]. The ratio of 4 ml acetone for every gram of PMMA was added to the feedstock as it could reduce the high shear rate and viscosity which would in turn enhance the mixing results of the feedstock [16]. The chemical composition of SS 316L is stated in Table 1 and the irregular shape of the particles is shown in Figure 1. Details of the PEG, PMMA and SA are shown in Table 2.

Table 1: Chemical composition of SS 316 L powder.

Elements	C	Si	Mn	P	S	Ni	Cr	Mo	Cu
Mass (wt.%)	0.027	0.84	0.19	0.016	0.012	12.20	16.40	2.10	0.03

Table 2 : Type of binder used for MIM process.

Binder	**Designation**	**Melting temperature,°C**	**Density, g/cm^3**
Component 1	Polyethelena Glycol (PEG)	63.32	1.23
Component 2	Polymethyl Methacrilate (PMMA)	257.77	1.19
Component 3	Strearic acid (SA)	70.1	0.94

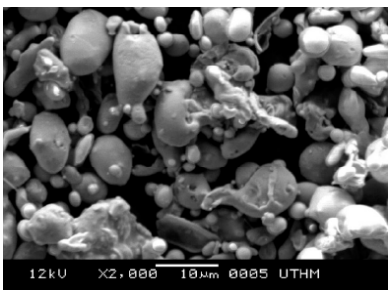

Figure 1: SEM image of SS 316L water atomised powder at 2000 x.

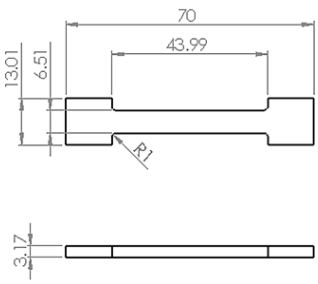

Figure 2: Tensile shape for MIM fabricated compact (mm).

The feedstock was prepared using Brabender plastograph EC rotary mixer with a rotation frequency of 26 rpm for 60 minutes at a temperature of 70 °C. The temperature of 70 °C is the mixing temperature within the highest melting temperatures and the lowest degradation temperature of binders for 73 vol. % PEG, 25 vol. % PMMA, and 2 vol% SA [17]. After mixing, the paste was removed from the mixer and left at room temperature and subsequently fed into a strong crusher to produce homogenised granules of feedstock. The compact tensile shape with diameter as illustrated in Figure 2 was introduced by injection moulding machine using optimal parameters. Next, the solvent debinding process was conducted using a water leaching process as the PEG is a water-soluble binder [18-22]. The compact was immersed in distilled water at five different temperatures: 40, 50, 60, 70, and 80 °C up to 12 h to observe the percentage of PEG loss from the compact. The debound compacts were dried for 1 h in the oven after every 2 h of being immersed in water. The purpose is to evaporate water from the pores before weighing the compact to observe the PEG loss for every 2 h up to 12 h.

3. RESULT AND DISCUSSION

Figure 3 shows the homogenised granules in pellet form as a result of the granulation process. After numerous trials, the feedstock which consisted of a PEG/PMMA/SA binder system was successfully moulded at a temperature of 215 °C. Table 3 shows the optimum parameter of injection moulding to fabricate the green compact. Figure 4a shows the completed compact using parameters from Table 3. Figure 4b shows the incomplete green compact injected at a temperature below 215 °C, which ranged between 120 to 210 °C. All green compacts in this study were found to have a short shot when injected below 215 °C. The surface morphology of the green compact with an injection temperature of 215 °C is shown in Figure 5 via optical microscopy (OM) at a magnification of 500 x. The OM image showed that the binder filled practically all the interstitial spaces between the powder particles. It also revealed that there were some voids in between the powder particles, which indicated that either air was entrapped, or that shrinkage of the binder occurred during cooling. The binder was distributed in a fairly uniform manner throughout the body.

Figure 3: The feedstock of SS 316L in this study.

Table 3: Optimum parameters for a complete injected green compact.

Injection temperature (°C)	Injection time (sec.)	Clamping time (sec.)	Cooling time (sec)
215	5	180	60

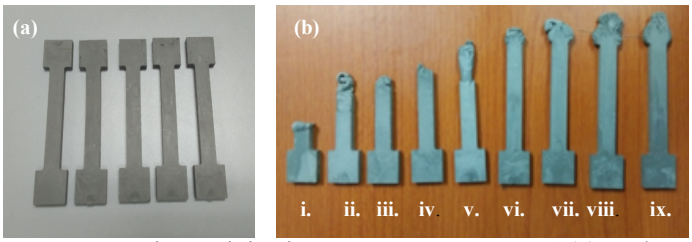

Figure 4 (a-b): Green compact using an injection temperature at 215 °C (a), and green compact using injection temperatures below 215 °C [at 120 °C (i), 150 °C (ii), 180 °C (iii, iv), 200 °C (v, vi), 205 °C (vii), 208 °C (viii), and 210 °C (ix)] (b).

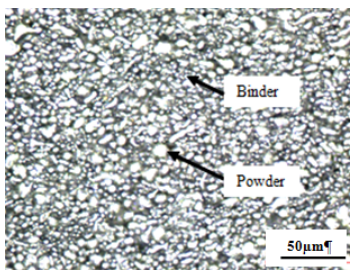

Figure 5: Microstructure of green compact via OM at 500 x.

The weight loss percentage of PEG was calculated according to the following equation,

$$\% \, Wt_{loss} = (W_{initial} - W_{after}) / W_{initial} \times 100 \quad [17] \tag{1}$$

where $W_{initial}$ is the weight of the green compact and W_{after} is the weight of the brown compact. During immersion, the water diffuses into the binder to react with the PEG and then dissolves the PEG binder. Figure 6 shows the kinetics of PEG removal at five different immersion temperatures: 40, 50, 60, 70 and 80 °C after 12 h. The weight of the debound sample was calculated every 2 h after being immersed in water and dried for 1 h to observe the amount of PEG loss. After 12 h being immersed in water, the debound sample at each immersion temperature was measured. Table 4 shows the weight loss percentage of the PEG. It was observed that the highest temperature which was 80 °C had the fastest rate of immersion of the PEG i.e. 100 % loss as compared to other immersion temperatures. This was also proven in previous studies [21, 23, 24], that the rate of removal of PEG was enhanced substantially by increasing the water temperature. Results from OM as shown in Figure 7(a-e) confirmed that as the time for debinding increased, the shape of the pores and particles became clearer with the remaining binder component of PMMA polymer attached to the particles as most of the PEG had been removed from the green compact.

Table 4: Percentage of weight loss at immersion times of 40, 50, 60, 70, and 80 ºC after 12 h.

Temperature (°C)	40	50	60	70	80
PEG loss after 12 h (%)	69	87	72	64	100

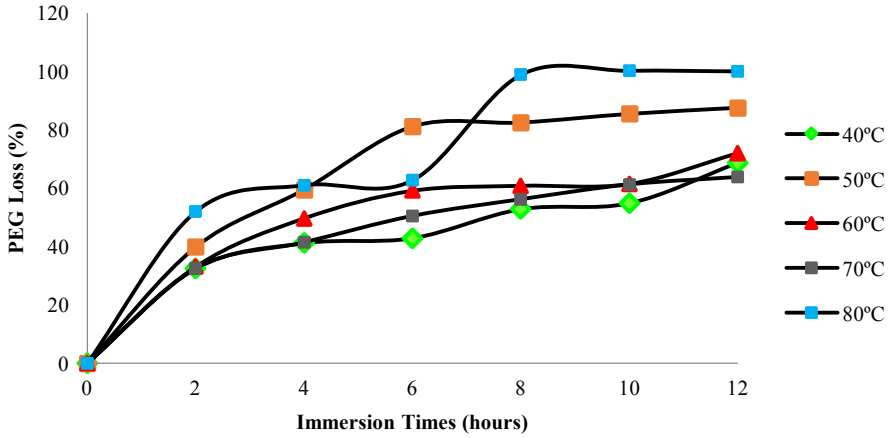

Figure 6: Kinetics of PEG removal at different immersion temperatures after 12 h.

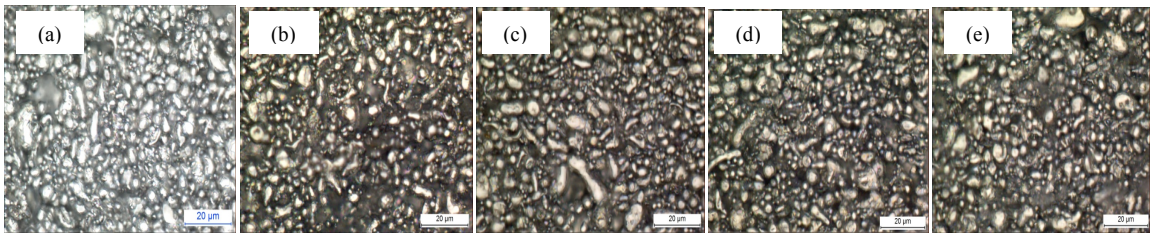

Figure 7(a-e): Optical microscope of interconnected pores development during solvent extraction of PEG removal at a magnification of 1000 after 12 h being immersed at various temperatures: (a) at 40 ºC, (b) 50 ºC, (c) 60 ºC, (d) 70 ºC, and (e) 80 ºC.

The homogeneity of the green compact and the brown compact using PEG/PMMA/SA binder system was analysed via density measurement using Archimedes principles according to MPIF Standard 42. Figure 8 shows the relative density results of five samples of a green compact with the same composition and the relative density of a brown compact at 40, 50, 60, 70, and 80 ºC after 12 h of being immersed in water. It can be observed that there was a small variation in density value, because of the difficulty to produce a homogeneously perfect feedstock [25]. The relative density of the brown compact shown in Figure 8 proved that the PEG was removed from the compact as the relative density values were decreased from the initial values before solvent debinding. The immersion temperature at 80 ºC demonstrated the lowest relative density as the PEG

loss was 100 % at this temperature after 12 h of being immersed in water. This was because the percentage of PEG loss was higher at a higher immersion temperature.

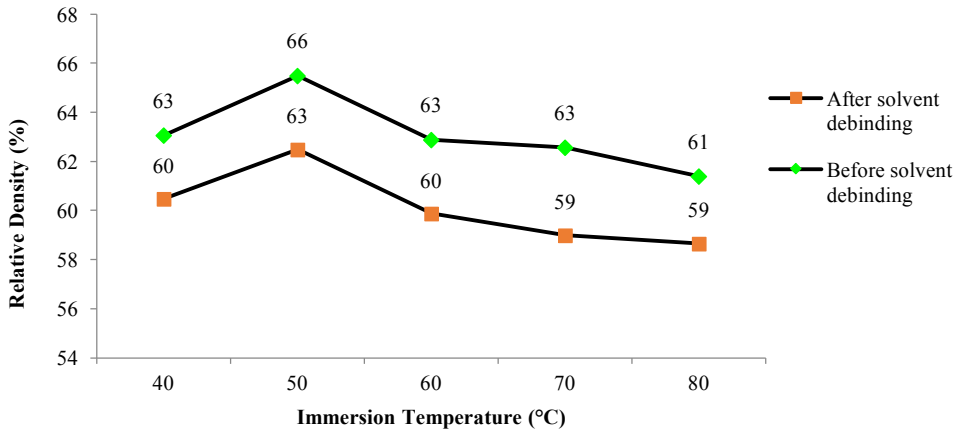

Figure 8: Relative density results of compact before and after debinding.

3. CONCLUSION

In summary, feedstock processability which combined water atomised SS 316L with a composite binder of water soluble PEG, with PMMA and SA were successfully injected through the MIM process. The 61 vol. % powder loading of SS 316L and binder formulations were mixed. Feedstock was utilised with 73 vol. % PEG, 25 vol. % PMMA and 2 vol. % SA, respectively. It was verified by the green compact density as a constant. Moreover, observations from the optical micrograph showed that the powder particles dispersed homogeneously in the matrix. Observations of PEG removal at five different immersion temperatures: 40, 50, 60, 70, and 80 ºC for up to 12 h of being immersed in water was studied. The percentage of PEG loss was calculated every 2 h up to 12h to investigate the percentage of PEG loss from the brown compact. After 12 h, the brown compact at the highest immersion temperature of 80 ºC showed 100% PEG loss. The removal of PEG was confirmed by the image from the optical microscope showing the shape of the pores and powder particles with PMMA polymer attached. Moreover, the relative density values of the brown compact were reduced when compared to the green compact which indicated the removal of PEG during the solvent debinding process. The lowest value of relative density was when the compact was immersed at 80 ºC as the PEG loss was 100 %.

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