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Effect of Immerse Temperature and Time on Solvent Debinding Process of Stainless Steel 316L Metal Injection Molding

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Abstract. This study was carried out to investigate solvent debinding by conducting the extraction process at temperature ranging from 40 to 80 ºC within 2 to 8 hours, while keeping the heptane solvent and 12:1 of solvent to feed ratio as constant. The palm kernel loss was evaluated as an indicator of the process's performance. It was also supported by the pore evolution that was observed by Field Emission Scanning Electron Micrograph (FESEM). Results show that both parameter give large effect on the solvent debinding performance. The best immerse temperature and time for extracting maximum palm kernel in heptane solution at S/F ratio of 12:1, without sacrificing the ability of producing free defect metal part were given by 80 ºC and 6 hours, respectively.

Introduction

Metal injection molding (MIM) process is drawing greatly attention for producing small and intricate part in high volume mass production [1-4]. Also, it was a versatile technology used for a variety of alloys and ferrous metals such as carbonyl iron, iron-nickel, various stainless steels, titanium, tungsten and tungsten heavy alloys, as well as, intermetallic compounds [5-7]. It becomes as a promising technology as it was producing the part with outstanding mechanical property such as strength, hardness and such on [8]. Generally, there are four process of this technology. First, the fine metal powder particles mixed with waxes and/or thermoplastic polymers to form a feedstock that can be molded. Next, the crushed feedstock is given desired shape using an injection molding machine. Then, the binders must be removed from the molded part without sacrificing the ability of retaining shape of the part, so called by debinding process. Lastly, the part is sintered at high temperatures, often to near theoretical densities [9].

However, among these processing steps, debinding process was presented as a critical stage which indicates the success of whole processes [8,10]. German & Bose 1997 reported the failure to remove

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most of the binders before sintering can result in part distortion such as cracking, swelling, warping and such on [5,11]. Also, Seeran et al. (2014) stated that the insufficient binder removal resulting in contamination from polymer decomposition during sintering [12]. Precisely, the effectiveness of solvent debinding process commonly based on the optimal debinding rate without compromising the part's quality [13]. Multiple steps of debinding process help to remove the binder without sacrificing shape retention of the metal compact was considered as a delicate step [8]. Often, debinding process conducted via solvent extraction and followed by thermal pyrolysis. This combination able to shorten the overall debinding cycle [14-16]. The open pore created during the solvent debinding causing the rapid loss of minor binder during thermal pyrolysis. Consequent with this, authors realize the important of critical study on the solvent debinding process need to be conducted, in purpose to deliberate the optimum performance of whole debinding process, followed by sintering.

The overall idea of solvent debinding was by taking the advantage of the high solubility of low molecular weight of binder (major binder constituent – 60 wt. % palm kernel) components in the organic solvent [8]. The green parts are immersed in a suitable organic fluid, which dissolves the binder partially. Many researchers studied different parameters that affecting the solvent debinding process. German and Bose (1997) reported that high powder loading required a long time of solvent debinding process [10]. It was due to the difficulties of the binder diffusion through the smaller pores. While, in another work, Zaky (2004) investigated the effects of different organic solvents (n-hexane, n-heptane, and isooctane) at different extraction temperatures (30, 40, 50, and 60 ºC) by varying the solvent to feed (S/F) ratios (ranging from 7:1 to 15:1 by weight) within extraction times (ranging from 1 to 5 hours) on the amount of binder extracted, diffusion coefficient of paraffin wax and the shape maintenance of green parts [10]. They stated that the dissolution of binders in suitable organic solvent is regularly used for accelerating the removal of the major constituents of the binder system while leaving the minor constituents to support the structure.

Thus, the objective of this present study was to investigate the effects of immersing temperature and time when extraction process conducted in heptane solution using 12:1 of S/F ratio on the palm kernel loss and pore channel built in the green parts. To be highlighted here, the both selections as a constant was made based on previous work by Zaky (2004) and supported by

Materials and methods

The metal powder used in this study was water atomized Stainless Steel 316L (purchased from Epson Atmix Corp) having irregular shape with mean size of $d_{50} = 6\mu m$. According to Ibrahim et al (2009), the critical powder volume concentration (CPVC) for this metal powder found to be approximately 64.80 % [6] . Relating to German & Bose (1997) statement, the optimum powder loading should be kept approximately 2-5% lower than critical loading [5]. Consequent with this, the appropriate powder loading of 60 vol. % was applied for this MIM study. Details characteristic of the powder and binder system are tabulated in table 1 and 2.

Table 1. SS316L powder characteristic [6].

| Characteristic	Details
Identification	SS316L, PF-10F
Tap density, g/cm^3	4.06
True Pcynometer density, g/cm^3	8.0471
Powder size	$d_{10}=2.87\mu m$
Identification	$d_{50}=5.96\mu m$
	$d_{90}=10.65\mu m$

a)   **Table 2. Summary of the binder's characterization [17].**

Binders	Palm kernel (PK)	Waste plastic bag (WPB)
Binder composition (wt. %)	60	40
Degradation temperature (ºC)	417.4365	473.94
Melting temperature (ºC)	35.125	124.25
Density (g/cm^3)	0.8852	1.0548
Source	Sime Darby	Recycle binder

The MIM process is performed following the diagram of figure 1. In this experiment, the MIM feedstock was 60 vol. % of water atomized Stainless Steel 316L (SS316L) powder mixed with binder system made up of; 60 wt. % of palm kernel (PK) and 40 wt. % of waste plastic bag (WPB). Interestingly, the waste plastic bag as a recycle binder was applied as recycle binder in order to replace the commercial binder of High Density Polyethylene which are quite expensive. This effort was so meant in reducing the disposal problem and encourage the "Green MIM" [18]. In another works of Ibrahim et al. (2016), this feedstock formulation was proven giving a good homogeneity of feedstock [17]. The materials are mixed by Plastograph Brabender mixing machine at 135 ºC for 80 minutes. In injection molding, the feedstock are molded into rectangular bar as shown in Figure 2 (a) using Horizontal screw injection molding machine. The injection molding was conducted at temperature of 180 ºC, mold temperature of 50 ºC, injection pressure of 40 % and injection speed of 50 %. The morphology of the green part was observed as presented in Figure 2 (b). It was used as a guidelines to observe the morphology's part, before and after solvent debinding. Figure shows the metal particles are well coated by palm kernel due to the homogenous feedstock used to produce the green compact [19].

The green parts were then subjected to a solvent extraction where the soluble binder of PK is leach out by immersing the part in heptane solution that ratio by 12:1 (S/F) at leaching temperatures of 40 to 80 ºC within 2 to 8 hours. Then, debound parts were dried at 50 ºC for 1 hour before the weight of the immerse part was taken. It was for evaporating the solvent from the pores. The debinding ratio (W_d) of PK were calculated based on weight percentage removed, and calculated by (1). Additionally, it was supported by the evolution of pore structure that was analyzed by Field Emission Scanning Electron Micrograph (FESEM).
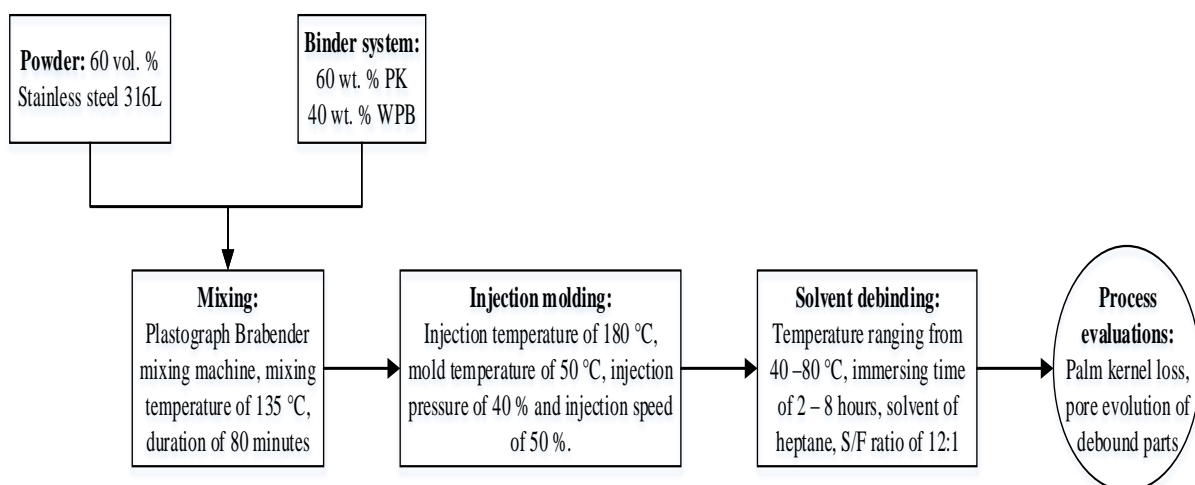
Figure 1. Diagram of the experimental procedures.
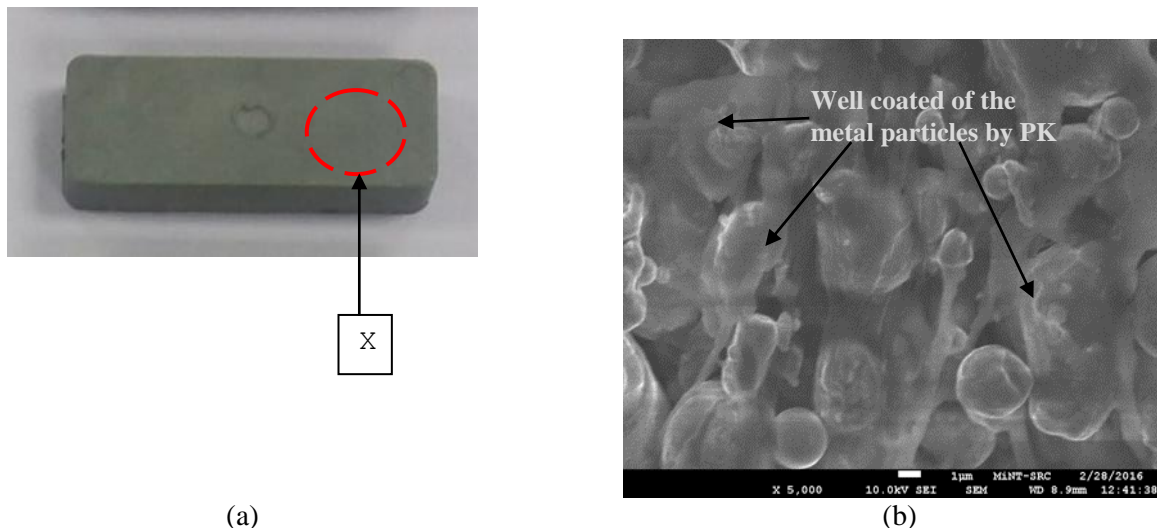
Figure 2. (a) Rectangular bar shape of green part produced, (b) Morphology of region X.

$$W_d\left(\%\right)=\frac{(W_i-W)}{W_i}\times100 \qquad (1)$$

Where W_i is the initial weight of compressed bodies and W is the weight after solvent debinding. The amount of binder extracted was calculated by dividing W_d by total binder content (wt. %) in the feedstock.

Result and discussion

Among many steps in the MIM, the solvent debinding process is considered the most important step. The suitable organic solvent is important for dissolving the binders and speed up the removal of the major constituents (PK) of the binder system while leaving the minor constituents (WPB) to support the structure. This minor constituents is removed later by a thermal debinding. The porosity network created by the solvent extraction step, make the removal of minor constituents of the binder system performed rapidly.

In this study, the solvent debinding process was carried out by extracting the green part in heptane solution at range temperature of 40 to 80 °C during 2 to 8 hours. Previous study by Zaky (2004) found that a very slight increase in the amount of binder extracted when applying an increase of solvent to feed (S/F) ratio [10]. Moreover, the cost of an extraction process is related directly to the quantity of solvent required for a given feedstock. Since that, by considering the economic point of view, a medium ratio of 12:1 was applied in this study, and acts a constant debinding variable.

Trend shows in Figure 3 was built in purpose to analyse the effects of debinding parameters on the solvent debinding process. The palm kernel loss during the process was measured by applying Equation (1) and it was act as an indicator for measuring the solvent debinding performance. Additionally, it supported by the pore evolution that was observed by Field Emission Scanning

Electron Micrograph. There are several important points that can be emphasized based on this built trend.
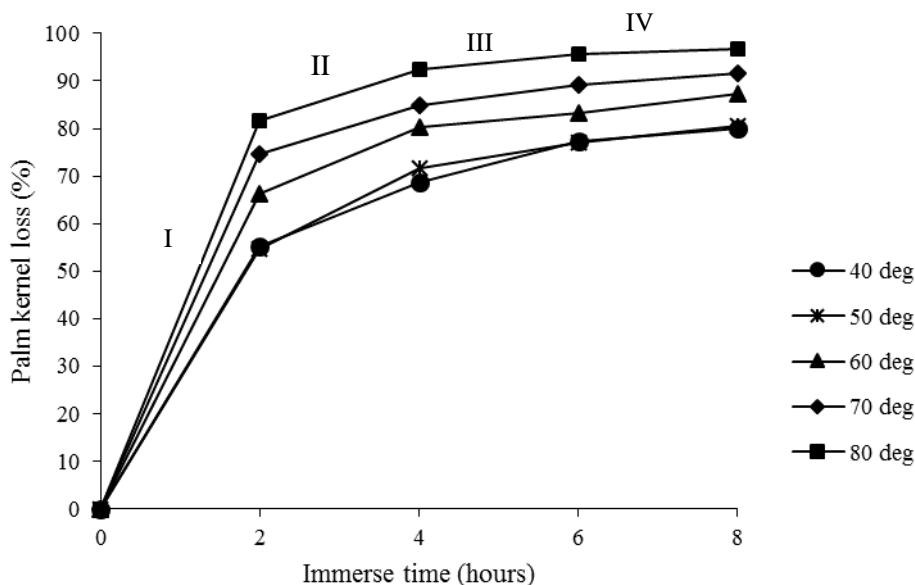
Figure 3. Trend of palm kernel loss at extraction temperature of 40, 50, 60, 70 and 80 °C when immersed in heptane solution for 2, 4, 6 and 8 hours of extraction as the 12:1 of S/F ratio used.

Firstly, the trend shows that the PK loss was increased as the extraction temperature increased. This was proven by the increase of binder loss when giving addition of extraction temperature by 10 °C. By referring to Figure 3, the binder loss for 8 hours of extraction at 40 °C (achieved palm kernel loss by 79.90 %), 50 °C (achieved palm kernel loss by 80.69 %), 60 °C (achieved palm kernel loss by 87.43 %), 70 °C (achieved palm kernel loss by 91.63 %) and 80 °C (achieved palm kernel loss by 96.71 %). So, it was about 16.81 % of PK loss obtained when increase the temperature by 40 °C. Meaning that, the average PK loss was valued by 0.42 % by adding up 1 °C of immerse temperature.

Other than that's, this fact was strongly supported by observing the pore evolution of the debound part produced when immersed in heptane solution at extraction temperature of 50 °C, 60 °C, 70 °C and 80 °C. Figure 4 shows the formation of the open pore channels as the palm kernel are extracted from the green part at different extraction temperature during 8 hours of extraction in heptane solution. The morphology structure for the solvent debinding focused on the voids created in an area dominated by a large group of binders. The existence of these pore channels within the particles facilitate the diffusion of minor constituent (WPB) of binder system during thermal debinding [12,20].

As can be seen in Figure 4 (a), (b), (c), (d) and Figure 4 (d), the higher extraction temperature create a larger void between the powder and binder particles since the greater value of binder loss recorded as the molecular chain moves quickly at the higher extraction temperature [20,21]. Therefore, the higher is the temperature, dissolution of the palm kernel from the part was increased. However, it must be remembered that a too fast removal of major constituents of binder can cause distortion [21].

It was proven that the higher PK loss was obtained as the higher temperature increased and created the larger pore channel. This result shows a good argument with Omar et al. (2003), Vielma et al. (2008), Harun et al. (2012), Chua et al. (2013), Seeran et al. (2014), Arifin et al. (2014), Ibrahim et al. (2016) and Suleiman et al. (2016) which were found that the higher extraction temperature was able to produce the larger pore channels and accelerates the binder loss [11-13,22-25].

Meanwhile, Chua et al. (2013) and Seeran et al. (2014) reported defects such as swelling, cracks, and etc. occurred at extraction temperature over than 60 °C [12,25]. This was due to the large void that

created between the powder and binder particles when the major constituent of binder system diffuse out from the green part. At the same time, a large amount of water entered through the existed void created. This phenomenon was affecting the bonding between the metal powders. Also, this phenomenon leads the part to swell. But, this was in contrast with present study, which was performing the solvent debinding over the extraction temperature of 60 ºC and produced free defect metal parts.

Also, by referring Figure 3, it was realized that the stage I give the rapid loss of palm kernel and it require explanation. Details of the stages were discussed as below [12-13,24]:

1) Stage I (0-2 hours): A dramatically increase of binder loss was occurred. It was due to the existing layer of a binder on the surface of the green compact. This layer was created as the metal particles are sheared during injection molding process. In this case, the surface layer of particle pack more densely and displace some binder to the surface. The layer eases the flow ability of the feedstock into the mold cavity. During solvent extraction, this surface layer can be hydrated and dissolved rapidly. In another words, the loss of the binder is very fast because the binder on the surface of the component is in direct contact with the solvent (slopes relatively very high).

2) Stage II (2-4 hours): As the debinding process continues, the pore channels extend to the inner region of the parts. The slope shows that the slow debinding rate was obtained in this stage. It was because the difficulties for the solvent to penetrate deeply into the part for dissolving and extracting the binder to the surface. The diffusion of dissolved binder to the surface of the component is facilitated by capillary forces which involve solvent extraction.

3) Stage III (4-6 hours): This stage still recorded an increase of PK loss. It was due to sufficient channels created within the metal particles which allows the diffusion of the dissolved PK to the surface.

4) Stages IV (6-8 hours): The debinding rate slows down and the mass loss remains constant (See the slopes). At this stage, a saturation point between the binder and solvent was reached.
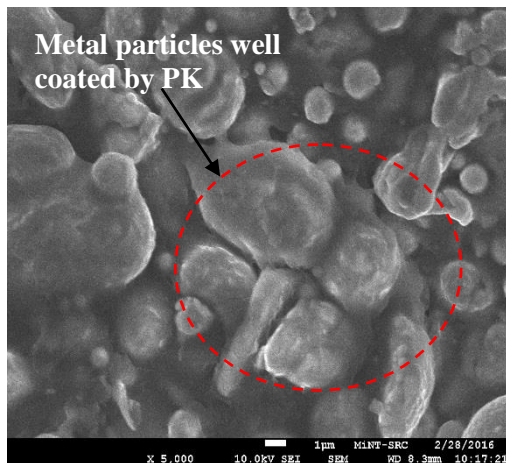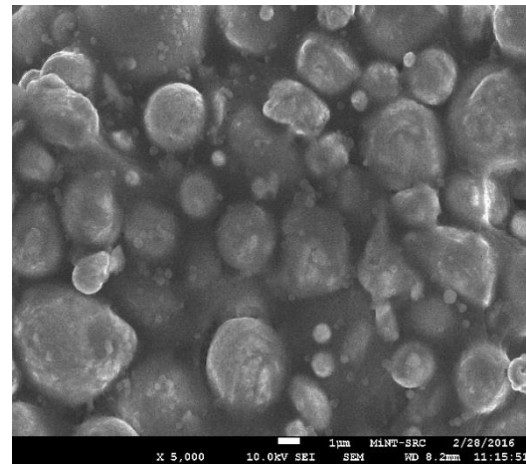
(a)   (b)
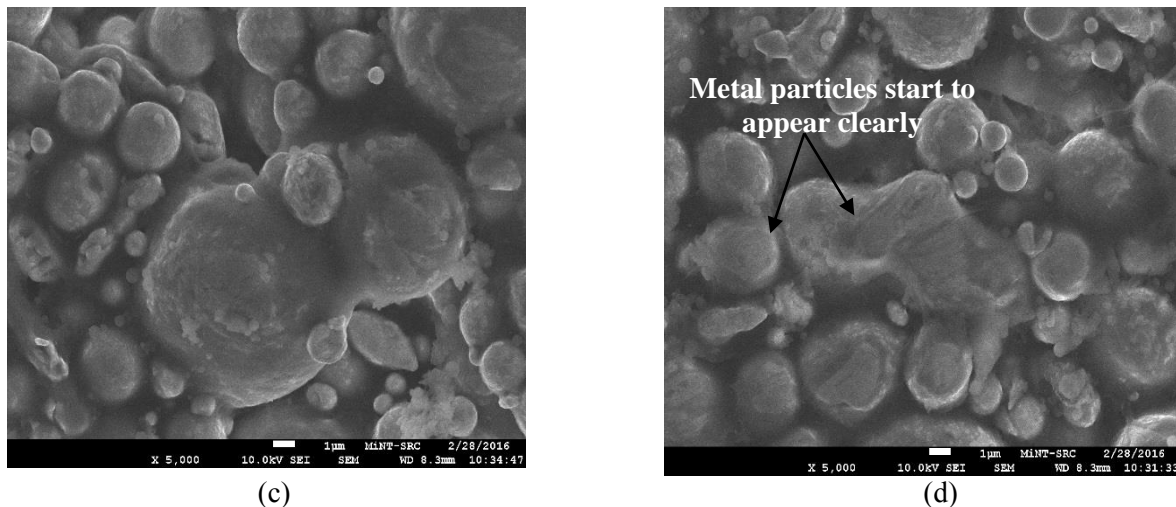
Figure 4. Open-pore evolution (magnification of x5000) of green part when immerse in heptane solution within 8 hours of extraction time at temperature of; (a) 40 °C, (b) 50 °C, (c) 60 °C, and (d) 70 °C.

German and Bose (1997) considered the time as important aspect in MIM [5]. The more time allocated for the process, the more cost required to be spent by the production line. Consequential of this, authors designed this present study in purpose to remove the major constituent of binder system in a short time with the minimum impact on the part [10]. Then, this investigation was continued by determining the best immerse time of the green part when extracted in heptane solution at 80 ℃ by also using 12:1 of S/F ratio.

Figure 5 presented development of the open pore channels after immersed in the heptane solution at 80 °C with given different extraction time which are 2, 4, 6 and 8 hours. The FESEM examination carried out on the solvent extracted parts indicated that all extractable organic portion of the binder was approximately removed after 6 to 8 hours of extraction. Noteworthy, the pore channels have formed in the green part after 2 hours of extraction. Figure 5 (a) clearly shows the large amount of palm kernel that coated all surface of metal particles when only immersed in 2 hours and it was supported by less value of binder loss, valued by 81.68 %. Also, it appears that the most of the waste plastic bag that links metal particles together are embedded within the palm kernel, in the necks of residual binder bridging the particles. As the extraction progresses, the removal of palm kernel in these regions reveals and exposes a network of strands waste plastic bag between the metal particles. It was clearly shown at the Figure 5 (d), the pore channels existed was largest compared to void created at the immersing time of 2, 4 and 6 hours. However, it can be noticed that there was only slightly increase of binder loss for 6 and 8 hours of immersion, valued by 95.77 % and 96.71 %, respectively. In other ways, only difference about 0.94 % for adding up the 2 hours of extraction, which is not worth to prolong the extraction duration.

Thus, by also considering the time limitation in manufacturing line [12], authors believed that the 6 hours of extraction time was the optimum time for conducting this solvent debinding process. This was strongly supported by the morphology of green part after 6 hours of extraction, as displayed in Figure 5 (c). It was shown that almost all of the palm kernel has been removed. A network of WPB ligaments remained, binding and holding the metal particles together to provide the MIM compact with sufficient brown strength to be handled [24]. Keeping in mind that the higher binder loss indicates by the larger size of pore channels formed. This was strongly supported by Omar et al. (2003), Vielma et al. (2008), Harun et al. (2012), Seeran et al. (2014) and Ibrahim et al. (2016) which stated that as extraction time increased, the weight loss of binder increased and the pore channels enlarged [8,12-13,22,24].
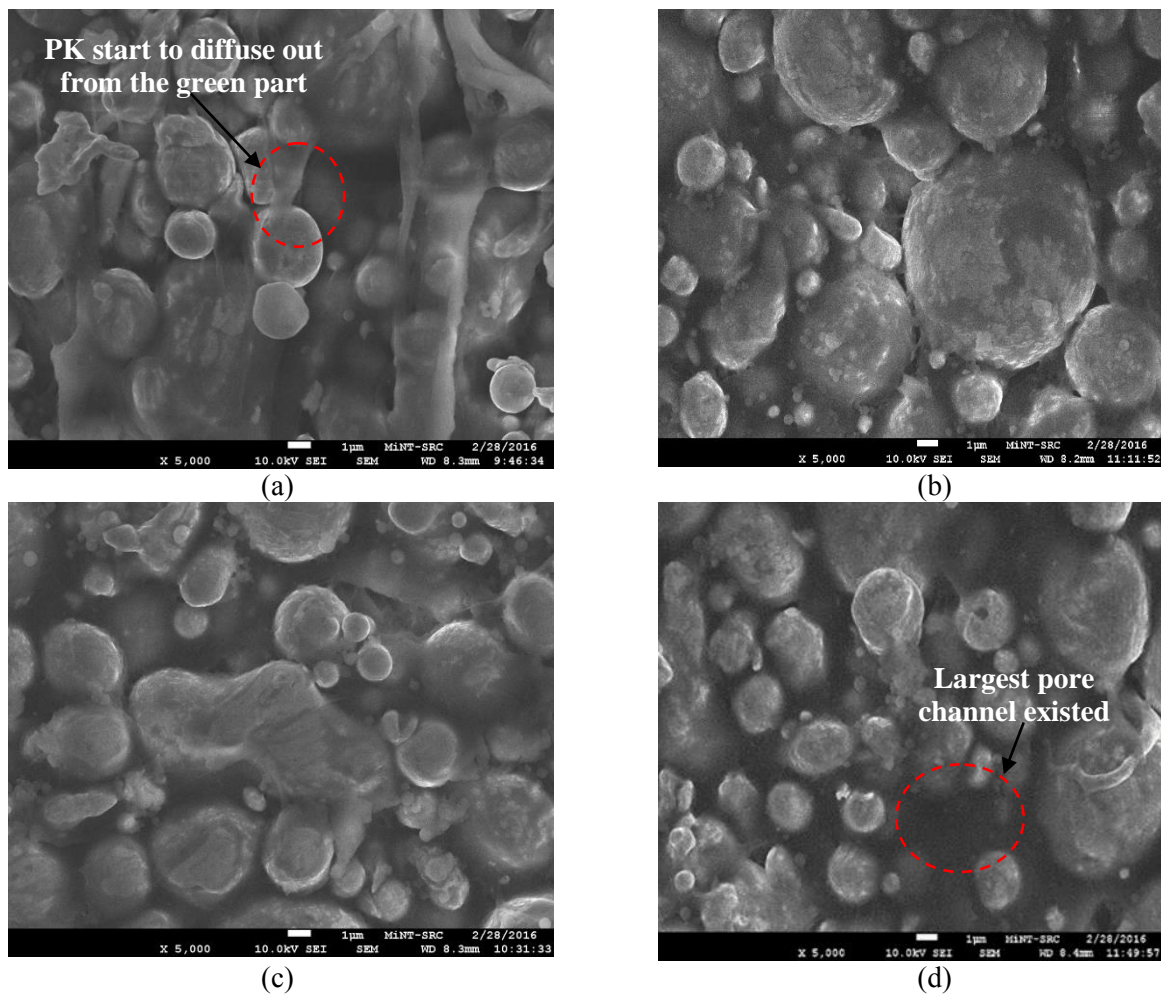
Figure 5. Open-pore evolution as a function of 80 °C of extraction temperature when immersed in heptane solution for; (a) 2 hours, (b) 4 hours, (c) 6 hours, (d) 8 hours.

During the solvent debinding process, all the specimens maintained their shape with no visible distortion and had adequate strength for handling. The remaining binder (WPB) is sufficient to hold particles in their positions. Thus, the remaining binder in both cases is sufficient to hold particles in their positions. Keeping in mind that, the residual binder can be removed rapidly during the thermal debinding process through the large pore channels existed.

Conclusion

The effect of immerse temperature and time on the solvent debinding process of palm kernel were investigated by measuring the percentage of binder loss, which supported by pore evolution observation. The findings are summarized as follows:

1) The higher is the extraction temperature, the higher PK loss obtained.
2) The longer is the immerse time, the higher PK loss recorded. But, the time limitation of manufacturing line need also to be considered.
3) The more binder loss, the larger pore channel existed in the debound part.

4) The optimum condition for the solvent debinding was conducted by immersing the green part in the heptane solution with 12:1 of S/F ratio at extraction temperature of 80 ºC within 6 hours.

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