

**MIXING AND RHEOLOGICAL CHARACTERIZATION OF WATER
ATOMISED STAINLESS STEEL POWDER FOR METAL INJECTION
MOULDING USING WASTE POLYSTYRENE AS BINDER SYSTEM**

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ABSTRACT

The issues to be highlighted here is to fundamentally evaluate the potential of using waste polystyrene as a backbone binder in Metal Injection Moulding (MIM.) This is by the fact that the earth has tons of polystyrene disposed every day and researcher believes such waste can be converted into more beneficial industrial product. Thus this research investigates the potential uses of polystyrene as a novel binder system in metal injection moulding. Stainless steel SS316L was used which is polystyrene and palm kernel acts as binder system. Feedstock with different composition 0.60% and 0.55% (60/40, 30/70, 20/80) powder loading (PL) were tested to know the feedstock characteristics. The homogeneity of the feedstock was investigated by using binder burn out test with Termogravimetic analysis (TGA). From the test it was found that PL 0.60% 70/30 is the best homogeneity. The rheology test was being held for better proven. The rheology properties are investigated using Shimadzu Flowtester CFT-500D capillary rheometer. The optimization of the μ MIM rheological properties as a function of stainless steel powder loading concentration are evaluated by flow behavior exponent, activation energy and mouldability index. The result show all the feedstock is a pseudo-plastic while no dilatant behaviour, it is no powder binder separation. Polystyrene are suitable acted as a binder system in metal injection moulding. Furthermore, it could reduce the cost of binder system. The feedstock achieved desirable injection moulding characteristics, such as homogeneous, stable, flow behaviour exponent, $n < 1$, moderate activation energy (E) and high mouldability index (α). The suitable volume ratios in mixing process produce a better feedstock in order to produce the green part by injection moulding.

ABSTRAK

Kajian ini menekankan tentang penggunaan sisa polisterina sebagai salah satu pengikat bagi proses pengacuan suntikan logam (*Metal injection moulding*). Hal ini berikutan keadaan bumi pada masa kini mempunyai pertambahan polisterina yang perlu dilupuskan setiap hari. Oleh itu, penyelidik percaya sisa itu boleh ditukar kepada bentuk produk industri yang berfaedah. Kajian ini menekankan tentang potensi polisterina bertindak sebagai agen pengikat dalam proses pengacuanan suntikan logam. Dalam kajian ini, keluli tahan karat (SS316L) digunakan sebagai serbuk logam manakala polisterina dan isirong sebagai bahan pengikat. Komposisi campuran bahan yang berlainan yang mana terdiri daripada 0.55% dan 0.60% (60/40, 30/70.20/80) pembebanan serbuk logam diuji bagi mengetahui ciri- ciri yang terdapat dalam campuran bahan. Kehomogenan campuran bahan telah diuji menggunakan ujian *binder burn-out*. Daripada ujian ini telah didapati bahawa pembebanan serbuk 0.60% 70/30 mempunyai sifat kehomogenan terbaik. Ujian reologi telah dijalankan untuk membuktikan lagi sifat homogen campuran bahan tersebut. Ciri – ciri campuran bahan telah diuji menggunakan Shimazu Flowtester CFT-500D Capillary Rheometer. Pengoptimumkan sifat reologi bagi keluli tahan karat, beban serbuk dinilai oleh index sifat aliran, tenaga pengaktifan dan kebolehcuan. Berdasarkan keputusan yang diperolehi semua campuran bahan menunjukkan sifat *pseudoplastic*, yang mana tidak ada sifat dilatant yang menyebabkan penyebaran serbuk pengikat semasa proses suntikan logam. Ciri- ciri acuan suntikan yang terbaik adalah seperti homogen, stabil index sifat aliran, $n < 1$ tenaga pengaktifan yang sederhana dan kebolehcuan yang tinggi. Polisterina sesuai digunakan sebagai bahan pengikat dan dapat mengurangkan kos bahan pengikat. Selain itu, nisbah jumlah yang sesuai dalam proses pencampuran mengeluarkan bahan mentah yang lebih baik untuk menghasilkan bahagian yang hijau dengan acuan suntikan.

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LIST OF SYMBOLS

$^{\circ}\text{C}$	-	Celsius
$^{\circ}\text{C}/\text{min}$	-	Celsius per minute
mm	-	Millimeter
μm	-	Micrometer
%		Percentages
α .	-	Moldability index
E	-	Activation energy
n	-	Flow behaviour exponent
mg	-	Milligram
kJ/mol	-	Joule per mole

LIST OF ABBREVIATION

<i>BSE</i>	-	Back Scattered Electron
<i>CPCV</i>	-	Critical Powder Volume Concentration
<i>DSC</i>	-	Differential Scanning Calorimetry
<i>EVA</i>	-	Ethylene Vinyl Acetate
<i>HDPE</i>	-	High Density Polyethylene
<i>LDPE</i>	-	Low Density Polyethylene
<i>MIM</i>	-	Material Injection Molding
<i>PP</i>	-	Polypropylene
<i>PW</i>	-	Paraffin Wax
<i>SA</i>	-	Stearic Acid
<i>SEM</i>	-	Scanning Electron Microscope
<i>SS316L</i>	-	Stainless Steel 316 Low Carbon
<i>STS 316</i>	-	Stainless Steel 316
<i>TGA</i>	-	Thermo Gravimetric Analysis
<i>DTA</i>	-	Differential Thermal Analysis
<i>PL</i>		Powder Loading

CHAPTER 1

INTRODUCTION

1.1 Background

Metal injection moulding (MIM) is a newly developed technology to form complicated shape metals and alloys. Metal injection moulding (MIM) offers a manufacturing capability for producing complex shapes in large quantities. The process utilizes fine metal powders is typically less than 20 micrometers, which are custom formulated with a binder like various thermoplastics, waxes, and other materials into a feedstock which is granulated and then fed into a cavity or multiple cavities of a conventional injection moulding machine.

After the “green” component is removed, most of the binder is extracted by thermal or solvent processing and the rest is removed as the component is sintered know as solid-state diffused in a controlled-atmosphere furnace. The MIM process is very similar to plastic injection moulding and high-pressure die casting, and it can produce much the same shapes and configuration features. However, it is limited to relatively small, highly complex parts that otherwise would require extensive finish machining or assembly operations if made by any other metal-forming process.

Metal injection moulding (MIM) offers several advantages over other production technologies. The advantages of metal injection moulding (MIM) are cost-effective

manufacture of high volume complex parts and reduced production time compared with investment casting. It also can reduce the cost of manufacturing compare to the other process such as casting and machining. Furthermore, Metal injection moulding (MIM) processes lie in its capability to produce mechanical properties nearly equivalent to wrought materials, while being a net-shape process technology with good dimensional tolerance control.

Among the many steps in MIM, the suitable choice of a binder becomes crucial to determine the mechanical properties of the final product. As a result, there has been increasing interest in developing lower cost methods of processing technique and a binder. The polystyrene waste and palm kernel become a new binder in metal injection moulding (MIM).

Polystyrene (PS) is a very versatile thermoplastic that is available as general purpose, high impact, and highly specialized resins. It is manufactured by the addition polymerization of styrene monomer. However, use of additives that influence resin formulation makes it possible to have PS resin grades. PS products are available in the form of pellets, powder, and granules. (Chritoper et al., 2003)

The binder is a temporary material for homogeneously packing a powder into the desired shape and holding the particles in that shape until the beginning of sintering. The binder is mixed with the powder to form feedstock for moulding. Unfortunately, no binders are perfect, so selection of appropriate binders varies between situations. The binders used in MIM are usually compounds of several materials. However, the binders may be broadly classified in two groups, those based on wax/polymer or, polymer/polymer compound. The wax/polymer binders are composed of a high polymer, a wax (or waxes) and additives. The purpose of the polymer is to impart rigidity to the part when cold, while the wax reduces the viscosity of the binder and the additives reduce particle separation and segregation. The wax/polymer binders are eliminated from the moulded part in the debinding step by either, solvent extraction followed by thermal treatment, or by thermal debinding alone. It has been well established however; that when one or various binder components are leached out and the others get burnt the part preserves its shape and dimension well until the sintering. The reason is that the porosity created during leaching open paths allowing the gasses resulting from the

decomposition of the other components to escape out of the part during burnt out. In this way, the generation of internal stresses is reduced avoiding the deformation of the parts.

Many binder systems are used in industrial operations. Part of this variation reflects the subtle differences in powder characteristics and debinding techniques. A general classification shows at least five types of binders used in MIM, most of which are polymers. These are categorized as thermoplastic compounds, thermosetting compounds, water-based systems, gelation systems and inorganics. (German R.M et al., 1997) The binders based solely on polymers are not very common in industry and have received little attention by researches everywhere. The reason is that the MIM process originated as an extension of the older Ceramic Injection Moulding (CIM) process in which water soluble binders were infrequent.

This research is intended to understand how Waste Polystyrene (PS) is suitable as binder to produce micro part through metal injection Moulding. To accomplish this, the binders and feedstocks are studied in a variety of aspects including, mixing, rheological behaviour, injection moulding, debinding, sintering and microstructure. This study will cover the first two processes only which are mixing and rheology.

1.2 Problem Statement

Development of new binders has always been at the most interest of researchers and has led to improvements such as cost reduction and less environmental issues. To date, extensive research has been done by using natural resources binder but none of them focused in waste material. The issues to be highlighted here is to evaluate the potential of using waste Polystyrene (PS) as a backbone binder in MIM. This is by the fact that the earth has tons of PS disposed every day and researcher believes such waste can be converted into more beneficial industrial products. However the main hindrances are the wettability and particle bonding between metal powder and waste PS. Moreover the mouldability performance, compatibility issue and PS diffusion during thermal degradation are the major problem as it contains hydrocarbon chain with a phenyl group attached to every other carbon atom. In this study Stainless steel SS316L will be mixed with waste PS and minimal amount of natural resources binder to improve the compact

shape retention. Here some criteria such as compounding and rheological behavior of the binder will be investigated before focusing on injection moulding, debinding and sintering. Since Micro MIM is still a new develop process and therefore every step processes behaviour have to be identified and predicted from the early stage. Extensive research has been done by using natural resources binder but none of them focused in waste material. The issues to be emphasize is to know the suitability of using waste polystyrene and palm kernel as a backbone binder in MIM.

Nowadays, polystyrene made our life easier in many ways. Unfortunately they are often not disposed properly. Polystyrene packing materials that we used for material contains carcinogens that may bring harm to the health of an impression; it causes serious illnesses such as cancer and so on. Moreover, the former polystyrene should not be destroyed easily. If this problem cannot be solved with proper it may worsen human health in the future.

Hopefully with this research, the polystyrene waste could be develop as new type of binder and becomes a novel research in metal injection moulding (MIM).

1.3 Objectives of Study

The objectives of this study are:

- i. To determine the suitability of polystyrene waste and palm kernel as a binder system in MIM.
- ii. To investigate the mixing and homogeneity of feedstock.
- iii. To investigate the rheological properties in order to get constant/steady viscosity graph (η -t).

1.4 Scope of Study

The scopes of this study are follows:

- i. Polystyrene and Palm Kernel are selected as binder and Stainless Steel (SS316L) act as metal powder.
- ii. Investigating the mixing procedure SS 316 L, Polystyrene and Palm Kernel.
- iii. Investigating the rheology behaviour at the different composition it is 0.55% (60/40, 70/30, 80/20) and 0.60% (60/40, 70/30, 80/20) of powder loading before being used in injection moulding.

1.5 Significance of Study

Nowadays, there were various types of food containers in market that have its own criteria. Each of them made from different materials with different specialty. At the meantime, these containers have invented through times in order to find a suitable container that could ease human's life. Some of them were made of polystyrene because of certain advantage that polystyrene have; such as light and disposable. Unfortunately these polystyrene not exactly disposed in other words even these material have advantages but it need long time to fully dispose. It is better to recycle these materials into other beneficial goods rather than it turns into harms. For instance it can cause harm to our environment and serious disease like cancer.

Polystyrene is known as a strong plastic that can be injected extruded or blow moulded. It is created from ethylene and benzene, where the thermoplastic polymer consists of long chain ethylene monomer. It is a very useful and versatile manufacturing material which it can make a lot of product such as container, household items, building material and many more. Normally standard polystyrene can easily be found in the market but it may cost at high price. As an option, this study is an effort to find other solution that can use wasted polystyrene food container as to replace it with standard polystyrene.

Therefore a new study was made in order to analyse the use of waste polystyrene in metal injection moulding (MIM) as a binder so that waste polystyrene food container could be recycled. Additionally, polystyrene waste and palm kernel are used as binders in this study because of its low cost and availability

CHAPTER 2

LITERATURE REVIEW

Literature review is major information of seeking a high quality research development in a particular field of study. This chapter reviews comprehensively about suitability of polystyrene waste and palm kernel as a binder system and stainless steel SS316L as metal powder. Besides that, this chapter also reviews on process during mixing and rheology process.

2.1 Introduction

In recent years, continuous effort of looking into the production of intricate metal components at low cost has led to the development of an innovative and cost efficient manufacturing process known as metal injection moulding (MIM). This manufacturing technique involves the combination of well-known plastic injection moulding technology with that the powder metallurgy (PM). Metal injection moulding are includes in four basic steps which is mixing, moulding, debinding and sintering. This fact is supported by Jamaludin et al., (2009). In this chapter, comparison from previous studies made the research more proper. From the previous study also can give a new knowledge for new researcher. Basically regarding with this reading also, it will ensure that the research was different with others.

Firstly, several journals were reviewed. The journal papers were obtained from variable publication. The selections of reading materials focus on process of metal injection moulding (MIM) and the possibility but the other relevant information also were reconsidered.

The reading materials related to MIM are review such as Mohd Afian Omar et al., (2008) concern on the influence of palm stearin content in the binder formulation based on rheological behaviour and various mixing conditions. Furthermore researches by Vivian et al., (2005) study the rheological of alumina moulding feedstock.

Besides that, a previous study by Liu et al., (2001) is focused on establishing a suitable binder system for MIM technology. This binder is useful as gaining information for new researcher. Other than that, research by Thian et al., (2002) is combining the productivity of injection moulding with the versatility of sintering of metal particulates. The feedstock preparation for MIM is a crucial step since deficiencies in quality of the feedstock cannot be corrected by subsequent processing adjustment.

Liu et al., (2005) were studies about the mixing and characterization of 316L stainless steel feedstock for micro powder injection moulding. Basically this study are investigated the effects of powder loading and extrusion on mixing besides the feedstock homogeneity.

Other than that, according to Paison et al., (2008) study about polystyrene is an inexpensive and rigid plastic. It is known as a light-weight material and has very good insulation properties. As a result, due to the fact that tons of waste PS disposed on earth every day, it attracts researcher in investigating the potential/novel binder with local natural resources and environmentally friendly.

According to M.H.I et al., (2008) were studies about rheological characteristic. In this study, stainless steel SS316L powder was mixed with composite binder, it is PEG (Polyethylene Glycol), PMMA (Polymethyl Methacrilate) and SA (Stearic Acid) by variation powder loading. Besides, the goals of this research are to develop a suitability feedstock for injection moulding process.

2.2 Powder Metallurgy

Metal powder is one of powder metallurgy concept. Hartwig et al., (1998) concluded that powder metallurgy is the process of blending fine powdered material and then heating the compressed material in a controlled atmosphere to bond the material. Although, compacting is generally performed at room temperature, and the elevated-temperature process of sintering is usually conducted at atmospheric pressure. Optional secondary processing often follows to obtain special properties or enhanced precision. Two main techniques used to form and consolidate the powder are sintering and metal injection moulding. There are many types of material such as stainless steel, tungsten heavy alloy, titanium, and intermetallic that is being used in metal injection moulding.

2.2.1 Stainless Steel 316L Metal Powder (SS316L)

In this present study, the chosen metal powder is stainless steel 316L in range of small particle. Referring to Paul et al., (2009) the number of 316 refers for the 3% of molybdenum and 16% nickel that added to the alloy iron, chromium and carbon.

The letter L is denotes to low carbon (<0.03%). Hence this metal powder also relatively biocompatible besides strong and cheap. Supported by Liu.L et al., (2005), 316L stainless steel powder was used since its good overall mechanical properties and corrosion resistance behavior are suitable for a wide range of applications. Small particle size and a multi-component binder system were used to meet the requirements for metal injection moulding (MIM).

Besides that, SS 316L is an austenitic Chromium-Nickel stainless steel with superior corrosion resistance. Therefore the particle size that use in this research is 5.71 μm and the density is 8.4071 g/m

Stainless steel powder is produced through water atomized or gas atomized powder. This study will focus on water atomized. Ibrahim et al., (2009) stated that water atomized stainless steel powder can provide good shape retention due to mechanical interlocking, its irregular shape particles yield relatively low packing density and exhibit

higher resistance to flow. The water atomization development efforts attempted to achieve near-spherical particle shape as a suitable compromise.

Hartwig et al., (1998) stated the advantage of the water atomized powder lies in its lower price. Also, the brown strength of parts produced with this kind of powder is fairly high as the irregular particles cannot flow past each other. However, the disadvantages of water atomized are the low tap density resulting in high sintering shrinkage as well as the tendency of the irregular particles to slightly align during injection moulding and thus causing anisotropic shrinkage. Figure 2.1 shown stainless steel 3316L morphological structure.

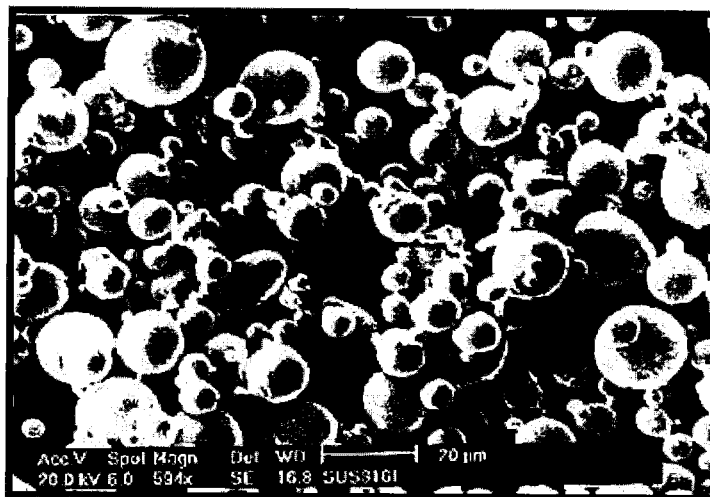


Figure 2.1: Stainless Steel 3316L Morphological Structure. (Khakbiz et al., 2005)

2.2.2 Chemical Composition of Stainless Steel

The chemical compositions of Stainless Steel as represented by ASTM A240 and ASME SA-240 specifications are indicated in the Table 2.1.

Table 2.1 Typical chemical properties for 316L grade stainless steels (Metals, 2004)

Grade		C	Mn	Si	P	S	Cr	Mo	Ni	N
316L	Min	-	-	-	-	-	16.0	2.00	10.00	-
	Max	0.03	2.0	0.75	0.045	0.03	18.0	3.00	14.0	0.10

2.2.3 Physical Properties of Stainless Steel

Table 2.2 Typical physical properties for 316L grade stainless steels (Metals, 2004)

Grade		316L
Density (kg/m ³)		8000
Elastic Modulus (Gpa)		193
Mean Co-eff of Thermal Expansion (µm/m/°C)	0 – 100 °C	15.9
	0 – 315 °C	16.2
	0 – 538 °C	17.5
Thermal Conductivity (w/m.K)	At 100 °C	16.3
	At 500 °C	21.5
Specific Heat 0 – 100 °C (J/kg. K)		500
Elec- resistivity (nΩ.m)		740

2.2.4 Mechanical Properties of Stainless Steel

Minimum mechanical properties for annealed Types 316, 316L, 317 and 317L austenitic stainless steel plate, sheet and strip as required by ASTM specifications A240 and ASME specification SA-240, are shown in Table 2.3.

Table 2.3 Typical physical properties for 316L grade stainless steels (Metals, 2004)

Grade	316L
Tensile Str (Mpa) min	485
Yield Str 0.2% proof (Mpa) min	170
Elong (% in 50mm) min	40
Hardness Rockwll B (HR B) max	95
Brinell (HB) max	217

The powder characteristics affect all subsequent processing decisions in metal injection moulding. A precise balance is needed between powder attributes, binder composition, and the ratio of powder to binder. In this study, there are concerns with the characteristics of the individual particles as well as with the collective (bulk) properties of a powder lot. Table 2.4 below shows the characteristic of 316L stainless steel analyzed by Lie et al.

Table 2.4: Characteristic of powder (Lie et al., 2008)

Material	Production Technology	Powder Shape	Average particle size/ μm	Tap density/ (g.cm^{-3})
316L	Water Atomization	Irregular	9.83	3.54
316L	Water Atomization	Irregular	17.83	3.81

Galio A.F. et al., (1997) investigated on 316L steel powder and found that the powder morphology and size distribution influence the mouldability of the feedstock and the quality of the final product. Furthermore, research by Hartwig et al., (1995) mention

that powder characterization is an important step for the production of high quality metal injection moulding parts.

2.3 Polystyrene

In this research, polystyrene are selected as a primary binder system due to the considerable of its own trade name which is styrofoam, a petroleum-based plastic which has been recognized worldwide environmental problem. It is known as a light-weight material and has very good insulation properties. Polystyrene basis is polymer and styrene which polymer derived from classical Greek poly means “many” and meres means “parts”. Schellenberg et al., (2009) was stated that styrene word defines the molecule contains double bond. Polystyrene is an inexpensive, rigid plastic and kind of vinyl polymer. Structurally, it is a long hydrocarbon chain with a phenyl group attached to every other carbon atom. Usually, polystyrene is produced by a radical or anionic polymerization starting from the monomer styrene. Generally, polystyrene is one of the items that will become as waste after used. Technically, polystyrene are produce from thermoplastic. Figure 2.2 shows the Polystyrene Molecule.

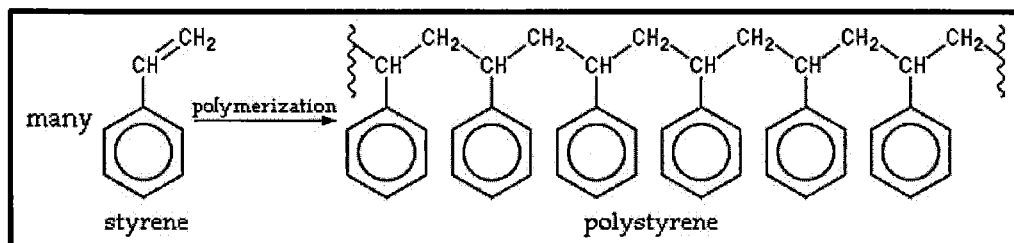


Figure 2.2: Polystyrene Molecule

Rigid polystyrene (PS) and related polystyrene plastic used as food packaging materials has had a longer history of use compare to others. Among the physical characteristics of the PS, there are some characteristics that have led to the development of other food plastic used in styrene for example, low impact strength and chemical resistance. Such features copolymerized with monomers such as butadiene and acrylonitrile, to provide flexible rubbery solid. In order to produce plastic food

packaging material there are several factors need to be considered such as physical characteristic of polymer, chemical structure, reactivity of monomer, volume of use of polymer in the industry, and known of toxicology of monomer. Table 2.5 shows the properties of styrene.

Table 2.5 Styrene Properties

PROPERTY	VALUE
Molecular weight	104.14
Boiling point, °C	145.2
Amorphous density	At 25°C: 1.05 g/cm ³
Glass transition temperature	100°C.

2.3.1 Polystyrene Food Container

Food packaging and other articles and materials that come into contact with food during storage, preparation, cooking and serving are a potential source of contamination. According to Dreck et al., (2004) were mention that chemical from polystyrene food container could leach from packaging into the food. And these might cause health effects from long term exposure, especially in sensitive consumers. This is an enormous variety of food packaging used, ranging from plastic and paper and board, which resins and inks, which may or may not be in direct contact with the food.

2.4 Palm Kernel

The secondary binder system that will be used in this research is palm kernel. Palm kernel was selected as secondary binder because its edible palm stearin and environmentally friendly with low toxicity and is harmless to humans. Normally, palm kernel is one part of the fruit of palm trees. Between all parts of the coconut palm, palm kernel seed only to be eaten. This fruit contains two variants oil palm oil obtained from the outside of the fruit while palm kernel oil is derived from the kernel itself. The temperature range is between 35 °C to 75 °C. The density in air of Malaysia Palm Kernel relies in range 0.9087 to 0.8809 g/ml. Hence, according to Majestic (2002) specific gravity for Palm Kernel is 0.90 - 0.92. Figure 2.3 shows the portion palm fruit.

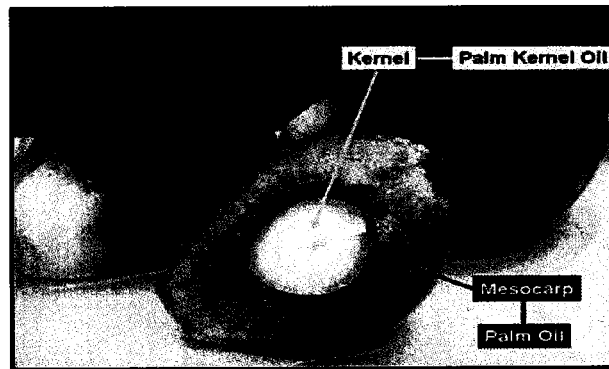


Figure 2.3: The Portion Palm Fruit

2.4.1 Chemical Characteristic of Palm Kernel Stearin

Table 2.6 shows the chemical characteristic of palm kernel stearin. Palm kernel stearin have been choose in this project because the content itself.

Table 2.6: Chemical properties of Palm Stearin (Porim technology, 1995)

Characteristics	Range	Mean	Std.dev
Iodine Value	5.8 to 8.1	7.0	0.51
Saponification value	245 to 254	248	4.08
Unsaponification matter % (weight)	0.22 to 0.60	0.32	0.18

2.5 Binder System

One of the major keys for successful MIM production is the binder system and its technology. Many different binders can be found in the literature, binders based on waxes are most common because wax-based binders are non-toxic, stable and easy to handle during mixing and moulding. The binder components used were Paraffin Wax (PW), Ethylene Vinyl Acetate (EVA) and High Density Polyethylene (HDPE). (Liu et al., 2011)

However, wax-based binders require a long debinding time at high temperatures. For large green parts, no matter how slow the heating rate during debinding, distortion of the part cannot be easily avoided. Another drawback of wax-based binders is separation of the wax and the polymer components during moulding because of low binder viscosity, incompatibility of the binder components, and poor adhesion of the wax to the powder.

The binder components were added in progressively, starting with the binder component with the highest melting point. According to Liu et al., (2001) the thermal properties of the binder components, the mixing temperature was set higher than the highest melting point, but lower than the lowest degradation temperature of the binder components.

2.5.1 Binder System Characteristic

Binder systems are act as temporary vehicle for homogenously packing the metal powder into a desired shape, besides it's also functions as packing agent in order to hold the particles in retention shape until the beginning of sintering process. (German et al., 1997) Binder systems are categorized in two types which is primary binder system and secondary binder system. Primary binder usually acts as packing agent and removes from the shape, hence the secondary binder were functions as a backbone while sintering process.

Tam et al., (1997) stated the characteristics of the binder have a large effect on parameters such as particle packing, agglomeration, mixing, rheology, moulding, debinding, dimensional accuracy, defects and final chemistry of MIM compacts. Good binders must possess the good flow characteristics, favorable interactions with the metal powder, good debinding characteristic, low cost; and be environmentally safe. Significantly, based on the previous research, the important guidelines could be used as a relevance reference as choose the binder system.

Apart from the major components, contemporary MIM binders usually contain additives, which improve flow properties of the feedstock, and increase the interaction between the metal and the binder. The MIM additives include dispersants, stabilizers, plasticizers, and inter-molecular lubricants. Dispersants, increase the wettability of the powder reducing the surface energy. Stabilizers, prevent particle agglomeration mostly by forming an adsorbed layer on the surface of the particles producing stearic stabilization. Table 2.7 shows some example binder used in Metal Injection Moulding (MIM)

Table 2.7: Components and properties of Binder

Material	Specific gravity (%)	Thermal conductivity joule.cm/(0°C cm ² / s)	Modulus, MPa
Copper	8.9	4.0	110.000
Steel (1040)	7.85	0.8	205.000
Concrete	2.4	0.01	14.00
Polyethylene (H.D)	0.96	0.0052	2.800
Polystyrene	1.05	0.0008	3.500
Polymethyl Methacrylate	1.2	0.002	3.500
Nylon	1.15	0.0025	3.800

2.6 Metal Injection Moulding (MIM) Process

A Metal Injection Moulding process involves several steps that combine both conventional Powder Metallurgy (P/M) and Plastic injection Moulding (PIM). Metal powder and binders are two main items needed in injection moulding. The powder used in this study is stainless steel 316L (SS316L) as this material is a highly alloyed material and the mechanical and corrosion properties are a concern (Ji et al., 2001). (Amin et al., 2001) Furthermore binders are one of the most critical factors in injection moulding. In this study waste food container Polystyrene and palm kernel were used as binders.

Metal injection moulding was the important method in this present study. Determination of suitable method depends on conditions of study need to emphasize to get valid results. Due to this, the method from previous study is very useful in new research. The methods that involve in previous study can be used as a guideline in this present research such as mixing, injection, debinding and sintering. Figure 2.4 shows flow chart of metal injection moulding.

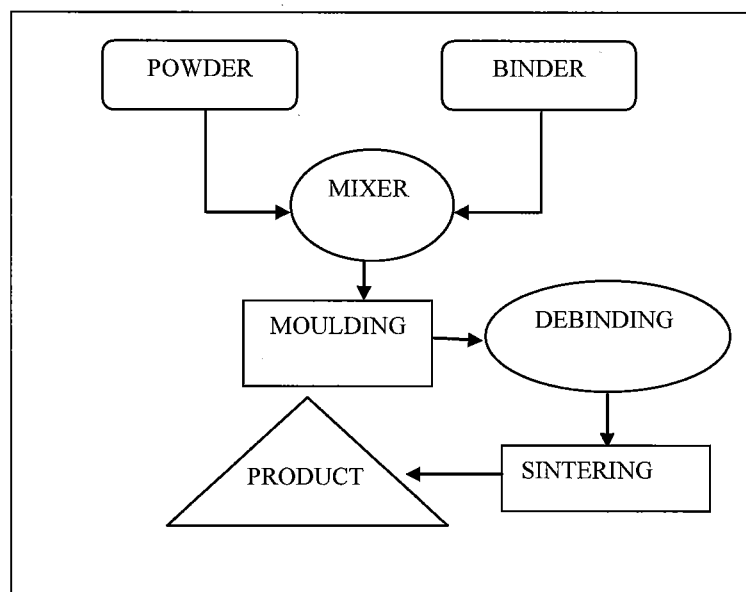


Figure 2.4 Flow chart of Metal Injection Moulding Process (Tam K.C et al., 1997)

2.6.1 Mixing

Mixing process is the important method in metal injection moulding in order to produce a quality compounding of feedstock. The temperature that was set while mixing process is between the melting point and lowest degradation of binders system. At this temperature, both of compounds between the metal powder and binder system will be mixed homogeneously. The mixing processes from the previous study are useful as a reference in establishing the method in this study.

The mixing process is one of the most important processes in metal injection moulding. Ibrahim et al., (2008) produced a quality feedstock, the mixing process between SS316 L, waste polystyrene and palm kernel stearin need to achieve a good homogeneity of mixture between metal powder and binder system. The homogeneity of a feedstock relates to how well the particulate solid is distributed in the binder matrix. Feedstock homogeneity promotes dimensional consistency of injected parts and helps preventing such defects as binder separation and powder segregation. To measure the homogeneity of a feedstock several methods are available including density measurements, binder burnt-out, capillary rheometry, and torque rheometry. From these four methods, the last two are reviewed.

There are both capillary and torque rheometers in use. They operate in different way. In capillary rheometers, the material is forced through a small gap channel from a reservoir by a piston. The pressure at the entrance of the capillary and the flow rate are recorded. On the other hand, torque rheometers are rotary equipments in which torque is recorded as a function of time of mixing.

In principle for determining feedstock homogeneity, separately from mixing studies, any rheometer should work. However, the capillary rheometers are preferred over any rotational types, such as parallel plates and cone and plate, because of three reasons. Capillary rheometers cover wider range of shear rates, the shear rates are closer to those created in injection moulding, and the flow conditions are closer to those found during mould filling. Therefore, the capillary rheometer reproduces the MIM processing conditions very closely. Torque rheometers, on the other hand, can be used to study the

whole mixing process, from the beginning of mixing to the end when the feedstock is produced. This is advantageous when a complete study of feedstock mixing is required.

2.6.2 Moulding

The moulding process involves heating the feedstock to a sufficiently high temperature where as it will melts, and then forcing it into a mould cavity to follow the shaped of the mould cavity. According to German and Bose (1997) mention the objective for moulding process is to attain the desired shape, free of defects with homogenous powder distributions.

The main part or process in MIM is injection moulding. The previous research can be used as a guide for injection moulding liked parameter, temperature and injection pressure. But temperature and pressure that will use change depend on the material that used to produce a feedstock.

Most of researchers (Supriadi *et al.*, 2007; Vielma *et al.*, 2008; Samantha *et al.*, 2011, Liu *et al.*,2001) who was involved in MIM area are conducted the experiment by using the injection mouldings method. The feedstock that has been cooled at a room temperature was feed in the injection moulding machine. The injection moulding parameter were optimized which is the temperature and the injection pressure. The barrel of injection moulding machine is heated and then the feedstock is melted inside the barrel. Injection is carried out above the upper melting point of the feedstock.

2.6.3 Debinding

Debinding are the next steps after the moulding process. Recently, there are several debinding techniques which are solvent and thermal process. Solvent extraction involves immersing the compact fluid that dissolves at least one binder phase, leaving an open pore structure for subsequent binder burnout. Another variant of solvent extraction is to heat the compact in the presence of a solvent vapor in a process akin to vapor decreasing (German and Bose, 1997). Debinding is listed as basic steps in this present research. The primary binder system will be removed from the part while the secondary binder system

remains in the part to retain the shapes. Most of all researchers used debinding techniques to remove the binder in their green part, and this section review the published research literature relate to debinding process.

Liu et al., (2001) conducted debinding process where multi component binder systems were used. The removal process of binder system are occurs at this stages. The remaining binder systems are acts to retain the shape of the part. In order to decompose the binder components, the green micro parts were heated with different heating rates and different holding time. The heating temperatures are near to the highest melting point of the binder.

According to Lin et al., (1998) the in situ length and temperature changes during debinding. The expansion of specimen was observed and influenced by the binder, solvent type and debinding temperature. When the debinding process not properly executed the part will cracking, distortion and slumping.

In the present research the debinding processes are useful to remove all the binder out from the moulded part. It is due to the multi component of binder system in the compounds. However at this stage the moulded part weight will be reduced. By using solvent debinding process the part will remains in rigid form without chemical reactions and open pore channels for subsequent easy degradation of binder. While by thermal techniques, there are no phase change and minimized defect formation on the parts.

2.6.4 Sintering

Sintering is a heat treatment process to bind the particles so that the material is in a solid state. Sintering method is necessary to remove all the binder from the part; the situation is to prevent part damage. The structure will shrink to a more compact and small. Sintering compaction generally occurs near the melting temperature of the material.

The type of sintering process and the sintering conditions are dependent on the composition and quantities of the materials to be sintered. The temperature is then increased up to the sintering temperature of the base alloy and maintained to ensure that solid state diffusion of the part occurs. The furnace and parts are then cooled. Depending

upon the application of component metallurgical requirements, cooling rates can be controlled to meet hardness and material density requirements.

Most of researchers (Supriadi *et al.*, 2007; Samantha.S.K *et al.*, 2011; Liu *et al.*, 2001) are conducted this steps in their research in order to obtain a better quality moulded part whereas good in mechanical properties and surface roughness. Sintering is the most important process in metal injection moulding. Figure 2.5 shown about sintering process flow chart (German and Bose 1997)

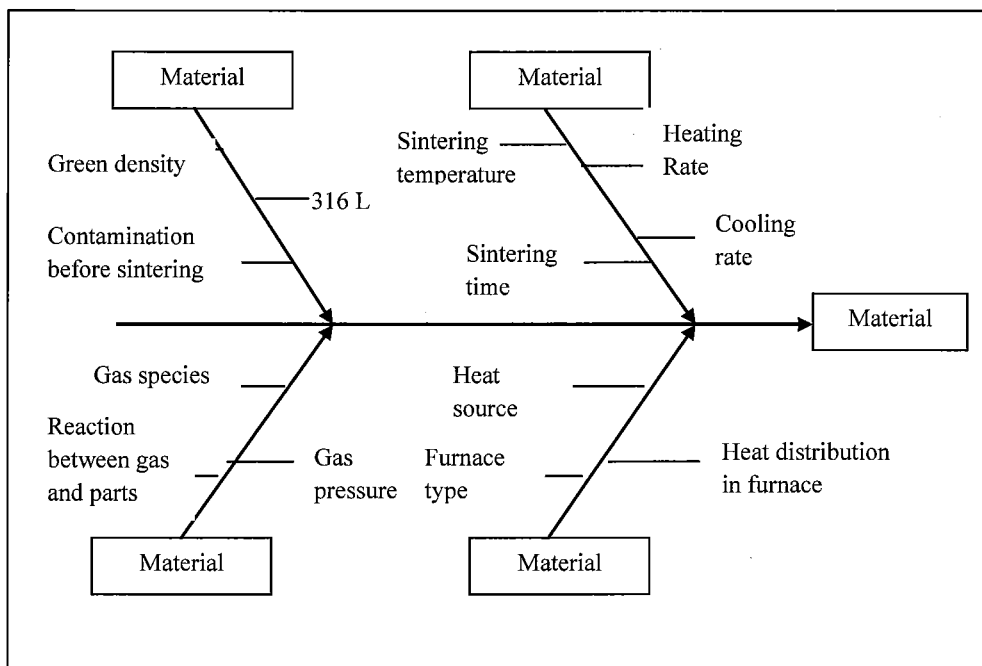


Figure 2.5: Sintering process flow chart (German.R and Bose, 1997)

2.7 Homogeneity Test

As mentioned before, the feedstock is the material that is moulded in MIM. Therefore, the properties of the feedstock determine largely the success of the whole MIM process. If the feedstock is homogeneous and stable, then the parts will have good qualities. Most properties of the feedstock will depend, then, on the compounding of the binder with the metal. The homogeneous and stable feedstock will be determined by homogeneity test.

The homogeneity of feedstock was assessed by comparing the weight loss (at the end of the testing) of five different samples. The samples were testing by Perkin-Elmer TGA.

Liu et al., (2005) stated that the feedstock with extrusion is best feedstock than the feedstock without extrusion. For the extruded feedstock, the particles disperse homogeneously into the matrix and are surrounded by the binder while according to Supriadi et al., (2007) were concluded that homogeneity of the feedstock corresponding with the mixing torque rates. At the low mixing torque, the feedstock more homogeneity compared to higher mixing torque since the material and metal powder are easy to mobilize.

2.8 Thermo Gravimetric Analysis (TGA)

Thermo Gravimetric Analysis (TGA) function to measures the mass change of a sample as a function of temperature or as a function of time. It is a method used for the characterization of the decomposition and the thermal stability of material under a variety of condition. The mass change characteristics are strongly depending on the experimental condition. There are some common curves obtained in thermo gravimetric measurements. Figure 2.6 shows the classification of TGA curves.

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