APPLICATION OF RESTAURANT WASTE LIPIDS (RWL) AS A BINDER COMPONENT IN METAL INJECTION MOULDING

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For my beloved mother PUAN HJH ZAMALIAH BTE SURATDI, my loving wife SITI NURFAIZAH BINTI MOHSAN, my sons MUHAMMAD AQEEL IFWAT, AHMAD AL-SHAREEF, my daughters KHAYRA ZAFIRAH, AREEFA NAFAESAH, my only sister and her husband INTAN KARTINI BTE MOHD AMIN, DR. HASMADI BIN HASSAN

“THANK YOU for your endless support”
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In the name of ALLAH S.W.T, The Most Gracious and The Most Merciful,

Greeting and Blessing to Prophet Muhammad S.A.W

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ABSTRACT

Application of Restaurant Waste Lipids (RWL) is introduced as binder component in metal injection moulding since it’s contains rich amounts of free fatty acids which is suitable as secondary binder components. Different binder formulation of RWL and Polypropylene (PP) were prepared as the binders and the mixture of these binders with water atomized 316L powder were obtained. The suitability application of RWL as binder component was monitored base on mixing condition, rheological characteristic, injection moulding, debinding and sintering process. Mixing time of 90 minutes was obtained as suitable mixing time for producing good homogenise feedstock base on mixing the polypropylene (PP) and RWL. Binder ratio of 50/50 weight percentage between PP and RWL was obtained to be good binder ratio although all binder ratio of 60/40, 40/60 and 30/70 shows pseudoplastic behavior. Taguchi method was successfully employed for optimizing the injection moulding parameters which consists of injection temperature, mould temperature, pressure, packing time, injection time, speed and cooling time. It was found that factors that contribute in injecting good part density and strength were temperature, pressure and speed. Extraction process of RWL using solvent debinding process indicates that hexane solution with temperature of 60°C and solvent to feeds ratio of 7:1 were better as compare to heptane with respect to fastest time removal. Good thermal debinding process under air atmosphere condition with temperature of 400°C and heating rate of 30°C/min was obtained. Sintering of the thermal debound parts also shows good mechanical properties and microstructure of 316L stainless steel parts.
ABSTRAK

Penggunaan sisa buangan lemak dan minyak dari restoran makan telah digunakan sebagai komponen bahan pengikat dalam pembuatan pembentukan suntikan logam. Nisbah percampuran yang berbeza antara RWL dan polimer polypropylene (PP) dihasilkan dan nisbah percampuran bahan pengikat yang berbeza ini dicampurkan bersama serbuk logam keluli tahan karat 316L. Keboleh bahan ini sebagai komponen bahan pengikat dalam menghasilkan bahan suapan serbuk logam 316L ditunjukkan dari segi percampuran sekata, sifat reologi, pembentukan suntikan, proses pembuangan bahan pengikat dan pensinteran. Masa adunan 90 minit menjadi pilihan berdasarkan campuran yang sekata antara bahan pengikat. Nisbah 50/50 berat bahan pengikat antara polimer dan minyak buangan dari restoran dipilih berdasarkan analisa yang dilakukan walaupun semua nisbah yang terdiri dari 60/40, 40/60 dan 30/70 didapat boleh digunakan sebagai nisbah bahan pengikat. Dalam mencari parameter acuan suntikan yang optimum bagi isipadu dan kekuatan komponen yang dihasilkan, kaedah rekabentuk eksperimen Taguhi digunakan dan mendapati bahawa faktor suhu suntikan, suhu acuan dan tekanan memainkan peranan utama bagi penghasilan komponen suntikan yang mempunyai ketumpatan dan kekuatan yang baik. Pengekstrakan bahan buangan lemak dan minyak dari komponen menggunakan pelarut hexane didapati lebih berkesan dari bahan pelarut heptane dengan suhu 60°C dengan nisbah berat pelarut dan komponen sebanyak 7:1 adalah lebih baik berdasarkan faktor singkatan masa. Pembuangan bahan pengikat polimer menggunakan kaedah haba dalam udara terbuka dengan suhu 400°C dengan kadar 30°C/min peningkatan suhu adalah lebih baik tanpa sebarang kecacatan pada komponen berlaku. Proses pensinteran terhadap bahan yang telah melalui proses pembuangan polimer menghasilkan pembentukan komponen yang baik dari segi sifat mekanikal dan mikrostruktur bahan 316L keluli tahan karat.
## CONTENTS

**DECLARATION OF THESIS STATUS**
**EXAMINERS’ DECLARATION**

<table>
<thead>
<tr>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>DECLARATION</td>
<td>i</td>
</tr>
<tr>
<td>DECLARATION</td>
<td>ii</td>
</tr>
<tr>
<td>DEDICATION</td>
<td>iii</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENT</td>
<td>iv</td>
</tr>
<tr>
<td>ABSTRACT</td>
<td>v</td>
</tr>
<tr>
<td>ABSTRAK</td>
<td>vi</td>
</tr>
<tr>
<td>CONTENTS</td>
<td>vii</td>
</tr>
<tr>
<td>LISTS OF TABLES</td>
<td>xiii</td>
</tr>
<tr>
<td>LISTS OF FIGURES</td>
<td>xvi</td>
</tr>
<tr>
<td>LISTS OF SYMBOLS AND ABBREVIATIONS</td>
<td>xxv</td>
</tr>
<tr>
<td>LISTS OF APPENDICES</td>
<td>xxviii</td>
</tr>
</tbody>
</table>

**CHAPTER ONE**  **INTRODUCTION**

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1 Problems Statement</td>
<td>4</td>
</tr>
<tr>
<td>1.2 Objectives</td>
<td>5</td>
</tr>
<tr>
<td>1.3 Scopes</td>
<td>6</td>
</tr>
<tr>
<td>1.4 Research Methodology</td>
<td>7</td>
</tr>
</tbody>
</table>

**CHAPTER TWO**  **LITERATURE REVIEW**

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.1 Powder Injection Moulding</td>
<td>9</td>
</tr>
<tr>
<td>2.2 Metal Injection Moulding</td>
<td>9</td>
</tr>
<tr>
<td>2.3 Binder</td>
<td>11</td>
</tr>
<tr>
<td>2.3.1 Backbone binder</td>
<td>12</td>
</tr>
<tr>
<td>2.3.1.1 Ethylene Vinyl Acetate (EVA)</td>
<td>13</td>
</tr>
</tbody>
</table>
2.3.1.2 Polyethylene (PE) 14
2.3.1.3 Polymethyl Methacrylate (PMMA) 14
2.3.1.4 Polypropylene (PP) 15
2.3.2 Secondary binder 16
2.3.2.1 Polyethylene Glycol (PEG) 16
2.3.2.2 Paraffin wax (PW) 17
2.3.2.3 Carnauba wax 18
2.3.2.4 Palm kernel and stearin 19
2.3.3 Surfactants 20
2.4 Restaurant waste lipids as binder (RWL) 21
2.5 Stainless steel 316L powder feedstock 24
2.5.1 Critical Powder Volume Concentration (CPVC) of SS316L powder 28
2.6 Feedstock formulation 31
2.6.1 Different binder components 32
2.6.2 Different weight or volume fraction of binder component 33
2.7 Mixing 34
2.7.1 Mixing rotational speed and time 35
2.7.2 Mixing temperature 37
2.8 Rheology 38
2.8.1 Flow behaviour index, n 41
2.8.2 Activation energy analysis, $E_a$ 44
2.8.3 Mouldability index, $\alpha_{STV}$ 46
2.9 Injection moulding 47
2.9.1 Factorial design (DOE) 49
2.9.2 Taguchi design (DOE) 50
2.10 Debinding 51
2.10.1 Single step thermal debinding process 52
2.10.2 Two step debinding via wicking and thermal debinding process 53
CHAPTER THREE  
METHODOLOGY

3.1 Introduction 67
3.2 Stainless steel powder used for feedstock 67
3.3 Binder components of feedstock 70
3.3.1 Polypropylene (PP) and Restaurant Waste Lipids (RWL) 70
3.4 Mixing 73
3.5 Rheology experimental method 76
3.6 Optimising green density and strength of the injection moulding processes 78
3.7 Solvent debinding process 82
3.8 Thermal debinding process 84
3.9 Sintering 87

CHAPTER FOUR  
RESULTS AND DISCUSSIONS: MIXING AND RHEOLOGICAL CHARACTERISTIC

4.1 Introduction 93
4.2 Characteristic of SS316L powder and binder components 93
4.2.1 Characteristic of SS316L powder 93
4.2.2 Characteristic of binder components 94
4.3 Mixing homogeneity
4.3.1 Homogeneity mixing time
4.3.2 Characteristic of mixing binder components
4.4 Effect of binder formulation on rheological behaviour and characterization of feedstock
4.4.1 Effects of temperature on viscosity with different binder formulation
4.4.2 Effect of binder formulation on activation energy, viscosity, flow behaviour index of feedstock
4.5 Optimising powder loading
4.5.1 Effect of powder loading to temperature variation with viscosity
4.5.2 Effect of powder loading on viscosity, activation energy, mouldability index and flow behaviour index of feedstock

CHAPTER FIVE
OPTIMISING INJECTION MOULDING
FOR DENSITY/STRENGTH OF GREEN PART

5.1 Introduction
5.2 Preliminary study of optimum density of injected parts (green part) for feedstock formulation of F1 with ISO 178 flexural bar shape
5.3 Optimising the green density of injection moulded tensile bar shape of F2 binder formulation
5.4 Optimising the F2 binder formulation for density and strength using L18 Taguchi method 147

5.5 Experimental Layout for Strength Measurement 152

CHAPTER SIX RESULT AND DISCUSSION:
DEBINDING AND SINTERING VARIABLES OF INJECTION MOULDED 316L WITH RWL AS BINDER

6.1 Introduction 156
6.2 Solvent Debinding 157

6.2.1 Effects of Solvents Temperature on Weight Loss Percentage of Green Compacts for F2 Feedstock During Solvent Debinding 157

6.2.2 Effects of Solvents Temperature on Diffusion Coefficient of Green Compacts for F2 Feedstock 160

6.2.3 Effect of Solvent to Feed Ratio on Solvent Debinding Time 164

6.2.4 Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS) on F2 Green Compact Formulation 166

6.3 Thermal Debinding 169

6.3.1 Effect of Temperature in Degradation of PP During Thermal Debinding 169

6.3.2 Effect of Heating/Cooling Rate on Weight Loss During Thermal Debinding 184

6.4 Sintering 185
6.4.1 Surface Appearance Between Green, After solvent, Brown and Sintered Part

6.4.2 Linear Shrinkage and Density of Sintered Part

6.4.3 Surface Morphology and Mechanical Properties of Sintered Part

CHAPTER SEVEN CONCLUSION

Conclusion

REFERENCES

APPENDIX
LIST OF TABLES

Table 2 - 1  Fatty acid composition (%) of NIE, EIE and CIE 50:50 PO:PKO blend fractions  19
Table 2 - 2  Reported fatty acids present in grease trap  22
Table 2 - 3  % mass fatty acids profiles of cooking fats and oils  23
Table 2 - 4  Fatty acid and FFA profile of FOG samples and neat palm oil  23
Table 2 - 5  Fatty acids composition of some vegetable oils and animal fats  24
Table 2 - 6  Various debinding technique for binder  61
Table 3 - 1  Chemical composition of water atomised SS316L (source of Epson Atmix Corp)  68
Table 3 - 2  Characteristic of water atomised SS316L  68
Table 3 - 3  Binder Formulation between PP and RWL with 60% powder loading SS316L  74
Table 3 - 4  Table of selected Injection parameter  80
Table 3 - 5  Injection Moulding Factors and its Levels using Taguchi L18  81
Table 3 - 6  Injection Moulding Factors and its Levels using Taguchi L27  82
Table 3 - 7  Temperature of solvent (Hexane and Heptane) used with time and temperature  84
Table 3 - 8  Experimental heating, cooling rate and temperature setup  86
Table 5 - 1  Injection parameters for optimised  130
Table 5 - 2  Binder components ratio and powder loading  131
Table 5 - 3 Level of Injection parameters for optimisation
Table 5 - 4 Experimental layout of the L16 and density measurement of the part
Table 5 - 5 Analysis of Mean and Signal to Noise Ratio of each factor
Table 5 - 6 Response table of variability analysis of S/N ratio
Table 5 - 7 Response Table for variability analysis of Mean
Table 5 - 8 Confirmation optimised injected part
Table 5 – 9 Injection Moulding Factors and its Levels
Table 5 - 10 Experimental layout of the L27 and density measurement of the part
Table 5 - 11 Analysis of Mean and Signal to Noise Ratio of each factor
Table 5 - 12 Response Table for variability analysis of S/N ratio
Table 5 - 13 Response Table for variability analysis of Mean
Table 5 - 14 Confirmation optimised injected part
Table 5 - 15 Injection Moulding Factors and its Levels
Table 5 - 16 Experimental layout of the L18 and density measurement of the part
Table 5 - 17 Analysis of Mean and Signal to Noise Ratio of each factor
Table 5 - 18 Average S/N ratio table
Table 5 - 19 Average Mean table
Table 5 - 20 Confirmation optimised density of green part
Table 5 - 21 Experimental layout of the L18 and strength measurement of the part
Table 5 - 22 Analysis of Mean and Signal to Noise Ratio of each factor
Table 5 - 23 Average S/N ratio table
Table 5 - 24 Average Mean ratio table
Table 5 - 25 Confirmation optimised strength of green part
| Table 6 - 1 | Carbon and Oxygen contents before and after solvent debinding process as compare to SS316L powder | 167 |
| Table 6 - 2 | Weight loss/gain after thermal debound with different temperature | 170 |
| Table 6 - 3 | Chemical composition of water atomised SS316L powder (Epson Atmix Corp.) | 172 |
| Table 6 - 4 | Shrinkage percentage after sintering process | 187 |
LIST OF FIGURES

Figure 1 - 1  Examples of MIM process sequence 4
Figure 2 - 1  Production of metal powder by gas atomisation, centrifugal and water atomisation 10
Figure 2 - 2  Examples of polymers used in different binder formulations 12
Figure 2 - 3  Mean concentrations of fatty acids in the FOG deposits 22
Figure 2 - 4  Powder particle shape of SS316L (a) water atomised (b) gas atomised 25
Figure 2 - 5  Critical powder loading by means of density measurement 26
Figure 2 - 6  Example of relative viscosity versus volume fraction at different shear rates 27
Figure 2 - 7  Mixing torque of finding critical solid loading metal powder 27
Figure 2 - 8  Example of powder particles shape 29
Figure 2 - 9  Brabender Plasti-Corder two screw mixer in open position 37
Figure 2 - 10 Typical (a) pseudoplastic, (b) Newtonian, (c) dilatant flow behaviour of fluid substances 39
Figure 2 - 11 Schematic diagram of capillary rheometer components 39
Figure 2 - 12 Examples of pseudoplastic (shear thinning), Newtonian and dilatant (shear thickening) flow behaviour on viscosity versus shear rate (force) profile 40
Figure 2 - 13 Examples of poor and well mixed of metal or ceramic powder and binder on capillary rheometer pressure profile 40
<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-14</td>
<td>Example of pseudoplastic behaviour of feedstock relationship between viscosity and shear rate</td>
</tr>
<tr>
<td>2-15</td>
<td>Relationship of flow behavior index ((n)) or power law exponent to temperature</td>
</tr>
<tr>
<td>2-16</td>
<td>Example of flow behavior with different particle size</td>
</tr>
<tr>
<td>2-17</td>
<td>Arrhenius-type plot for the OA, SA, and HSA suspensions; different slopes indicate the suspension viscosity exhibiting a different but specific temperature dependency</td>
</tr>
<tr>
<td>2-18</td>
<td>General moldability index vs. solid loading %</td>
</tr>
<tr>
<td>2-19</td>
<td>Moulding variables using factorial design</td>
</tr>
<tr>
<td>2-20</td>
<td>Effects of different heating rate on degradation temperature of binder components</td>
</tr>
<tr>
<td>3-1</td>
<td>Particles analyser machine (Fritsch analysette 22 compact)</td>
</tr>
<tr>
<td>3-2</td>
<td>JEOL JSM-6380LA Scanning Electron Microscopy</td>
</tr>
<tr>
<td>3-3</td>
<td>Critical powder volume concentration (CPVC) of SS316L powder</td>
</tr>
<tr>
<td>3-4</td>
<td>(a) Polypropylene (PP) and (b) RWL used in the investigation</td>
</tr>
<tr>
<td>3-5</td>
<td>Auto Q20 differential scanning calorimeter (DSC)</td>
</tr>
<tr>
<td>3-6</td>
<td>Linseis Thermal gravimetric Analysis Equipment</td>
</tr>
<tr>
<td>3-7</td>
<td>Perkin Elmer FTIR equipment</td>
</tr>
<tr>
<td>3-8</td>
<td>Mattler Toledo density apparatus measurement</td>
</tr>
<tr>
<td>3-9</td>
<td>Brabender Plastograph EC mixer is used for compounding SS316L powder, PP and RWL (b) RWL being heated using hot plate heater</td>
</tr>
<tr>
<td>3-10</td>
<td>Mixing sequence of feedstock compound using mixer a) PP into the mixing chamber, b) PP left for melting, c) SS316L powder left for homogeneously mix with PP, d) RWL going into chamber, e) feedstock being taken out, f) feedstock being cooled at room temperature</td>
</tr>
<tr>
<td>3-11</td>
<td>(a) Crusher machine used in transforming the bulky feedstock into (b) small pallet feedstock</td>
</tr>
</tbody>
</table>
Figure 3 - 12  Capillary rheometer used in determining the rheological behaviour and characterization  
77
Figure 3 - 13  Schematic diagram of capillary rheometer  
77
Figure 3 - 14  Injection moulding machine unit used for green specimens  
78
Figure 3 - 15  Mould of specimen ISO 178 flexural bar shape  
79
Figure 3 - 16  Mould of specimen MPIF Standard 41 bar shape  
80
Figure 3 - 17  Universal flexural/tensile machine  
81
Figure 3 - 18  Mould of specimen ISO 527-2 tensile bar shape  
82
Figure 3 - 19  Water bath used for solvent debinding process  
83
Figure 3 - 20  Colourless heptane change to yellow/brown colour during solvent debinding process of green compact  
84
Figure 3 - 21  High temperature furnace and temperature profile of thermal debinding process  
85
Figure 3 - 22  Alumina crucible  
85
Figure 3 - 23  X-ray diffraction Bruker D8 Advance machine  
87
Figure 3 - 24  Vacuum Furnace at AMREC Kulim, Kedah  
87
Figure 3 - 25  Mould sample holder  
88
Figure 3 - 26  Grit size sequence of polishing sintered SS316L part  
89
Figure 3 - 27  Grinding and polishing machine  
89
Figure 3 - 28  Silicon carbide grinding paper  
89
Figure 3 - 29  Polishing process  
90
Figure 3 - 30  Samples test after being grinded and polished  
90
Figure 3 - 31  Electro etching equipment setup for SS316L sintered part  
91
Figure 3 - 32  Microhardness test equipment  
91
Figure 3 - 33  Digital microscope used for determining the grain size  
92
Figure 3 - 34  Tensile machine test of the sintered part  
92
Figure 4 - 1  (a) Powder size distribution of SS316L, (b) SS316L powder morphology

Figure 4 - 2  Differential scanning calorimetry curve of RWL

Figure 4 - 3  Differential scanning calorimetry curve of PP

Figure 4 - 4  Thermal gravimetric analysis of the RWL

Figure 4 - 5  Thermal gravimetric analysis of the PP

Figure 4 - 6  Fourier Transform Infrared Spectrometry (FTIR) spectrum of RWL

Figure 4 - 7  Surface morphology of the feedstock with binder formulation (a) F1 (b) F2 (c) F3

Figure 4 - 8  TGA comparison of mixing time of Stainless steel powder 316L, RWL and PP for (a) 45 minutes (b) 90 minutes for F2 binder formulation

Figure 4 - 9  TGA profiles of different binder formulation

Figure 4 - 10  DSC of F1 relatives to PP and RWL derivatives

Figure 4 - 11  DSC of F2 relatives to PP and RWL derivatives

Figure 4 - 12  DSC of F3 relatives to PP and RWL derivatives

Figure 4 - 13  DSC of F4 relatives to PP and RWL derivatives

Figure 4 - 14  DSC scans of the PP, RWL and feedstock formulation

Figure 4 - 15  Viscosity versus shear rate graph of binder F1 with 60% powder loading

Figure 4 - 16  Viscosity versus shear rate graph of binder F2 with 60% powder loading

Figure 4 - 17  Viscosity versus shear rate graph of binder F3 with 60% powder loading

Figure 4 - 18  Viscosity versus shear rate graph of binder F4 with 60% powder loading

Figure 4 - 19  Viscosities shear rate of all binder formulations at temperature 190°C

Figure 4 - 20  Logarithmic viscosity against reciprocal temperature of F1 binder formulation at different shear rate
Figure 4 - 21 Logarithmic viscosity against reciprocal temperature of F2 binder formulation at different shear rate  
Figure 4 - 22 Logarithmic viscosity against reciprocal temperature of F3 binder formulation at different shear rate  
Figure 4 - 23 Logarithmic viscosity against reciprocal temperature of F4 binder formulation at different shear rate  
Figure 4 - 24 Comparison of logarithmic viscosity against reciprocal temperature of all binder formulation at different shear rate  
Figure 4 - 25 Activation Energy and flow behaviour index against binder formulation profile at shear rate of 1000s$^{-1}$  
Figure 4 - 26 Viscosity and flow behaviour index against binder formulation profile  
Figure 4 - 27 Activation energy and mouldability index against binder formulation profile  
Figure 4 - 28 General mouldability index vs. feedstock formulation  
Figure 4 - 29 Viscosity versus shear rate graph of binder F2 with 60% powder loading  
Figure 4 - 30 Viscosity versus shear rate graph of binder F2 with 61% powder loading  
Figure 4 - 31 Viscosity versus shear rate graph of binder F2 with 62% powder loading  
Figure 4 - 32 Viscosity versus shear rate graph at 190°C for all Powder loading  
Figure 4 - 33 Viscosity and flow behaviour index against Volumetric Powder Loading profile at 1000s$^{-1}$ shear rate  
Figure 4 - 34 Logarithmic viscosity against reciprocal temperature of 60% powder volume loading at different shear rate  
Figure 4 - 35 Logarithmic viscosity against reciprocal temperature of 61% powder volume loading at different shear rate  
Figure 4 - 36 Logarithmic viscosity against reciprocal temperature of 62% powder volume loading at different shear rate
<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>4 - 37</td>
<td>Comparison of logarithmic viscosity against reciprocal temperature of all powder volume loading at 1000 s(^{-1}) shear rate</td>
</tr>
<tr>
<td>4 - 38</td>
<td>Viscosity and activation energy against Volumetric Powder Loading profile at 190°C</td>
</tr>
<tr>
<td>4 - 39</td>
<td>Activation energy and flow behaviour index against Volumetric Powder Loading profile</td>
</tr>
<tr>
<td>4 - 40</td>
<td>Activation energy and mouldability index against Volumetric Powder Loading profile</td>
</tr>
<tr>
<td>4 - 41</td>
<td>Mouldability index and flow behaviour index against Volumetric Powder Loading profile</td>
</tr>
<tr>
<td>4 - 42</td>
<td>Viscosity and mouldability index against Volumetric Powder Loading profile</td>
</tr>
<tr>
<td>5 - 1</td>
<td>ISO 178 flexural bar shape</td>
</tr>
<tr>
<td>5 - 2</td>
<td>Schematic diagram dimension of ISO 178 flexural bar shape</td>
</tr>
<tr>
<td>5 - 3</td>
<td>S/N ratios variations of green part density at various levels of injection parameters</td>
</tr>
<tr>
<td>5 - 4</td>
<td>Mean variations of green part density at various levels of injection parameters</td>
</tr>
<tr>
<td>5 - 5</td>
<td>Variations of density during the L16 Taguchi DOE experiments</td>
</tr>
<tr>
<td>5 - 6</td>
<td>Injected part shape for solvent debinding (F2 feedstock)</td>
</tr>
<tr>
<td>5 - 7</td>
<td>S/N ratio of green part density at various levels of injection parameters</td>
</tr>
<tr>
<td>5 - 8</td>
<td>Mean of green part density at various levels of injection parameters</td>
</tr>
<tr>
<td>5 - 9</td>
<td>Tabulated density of green part density at various levels of injection parameters</td>
</tr>
<tr>
<td>5 - 10</td>
<td>Bar shape injected moulded part</td>
</tr>
<tr>
<td>5 - 11</td>
<td>S/N Ratio main effect plot of the control factors</td>
</tr>
<tr>
<td>5 - 12</td>
<td>Mean main effect plot of the control factors</td>
</tr>
</tbody>
</table>
Figure 5 - 13  S/N Ratio main effect plot of the control factors 154
Figure 5 - 14  Mean main effect plot of the control factors 154
Figure 6 - 1  Schematic diagram of solvent debinding process inside the green compact 156
Figure 6 - 2  Solubility of the RWL in (a) Hexane solution after 4 hrs (b) Heptane solution after 2 hrs with solvent temperature of 50°C respectively 157
Figure 6 - 3  Weight loss percentage of RWL removed for F2 feedstock during solvent debinding at different temperature using hexane 158
Figure 6 - 4  Weight loss percentage of RWL removed for F2 feedstock during solvent debinding at different temperature using heptane 159
Figure 6 - 5  Injected test specimen of the F2 feedstock (a) green compact (b) after extraction process 159
Figure 6 - 6  Effect of temperature on weight loss of the green compact during the first hour for F2 feedstock with different organic solvent 160
Figure 6 - 7  Diffusion coefficient of RWL for F2 feedstock during solvent debinding at different temperature using hexane 161
Figure 6 - 8  Diffusion coefficient of RWL for F2 feedstock during solvent debinding at different temperature using heptane 162
Figure 6 - 9  Effect of temperature on solvent debinding diffusion coefficient during the first hour for F2 green compact with different organic solvent 163
Figure 6 - 10  DSC profile of F2 green compact before and after first hour solvent debinding process using hexane at 60°C taken from the centre of the compact 163
Figure 6 - 11  Bar shape used for solvent debound process with different solvent to feed ratio at 60°C 164
Figure 6 - 12  Weight loss percentage profile of different solvent to feed ratio using hexane solution 165
Figure 6 - 13  Diffusion coefficient profile of different solvent to feed ratio using hexane solution 165
Figure 6 - 14 Evolution of pores from (a) green compact (b) solvent debinding after 3 hrs (c) solvent debinding after 6 hrs

Figure 6 - 15 EDS analysis on solvent debound sample (a) area taken for carbon analysis, (b) EDS element analysis of green samples, (c) after solvent samples, (d) after thermal samples

Figure 6 - 16 Comparison in dimension between brown part under different temperature

Figure 6 - 17 TGA profiles of brown compact after solvent debound process

Figure 6 - 18 XRD pattern of SS316L water atomised powder

Figure 6 - 19 Schaeffler diagram for determining phases formed upon solidification, based on chemistry

Figure 6 - 20 XRD pattern for (a) part with 500°C (b) part with 600°C thermal debound

Figure 6 - 21 XRD pattern of thermal debound part at 600°C indicates of oxidation of SS316L brown compact

Figure 6 - 22 Example of XRD profile oxidation occurs on SS316L powder during sintering under different atmosphere

Figure 6 - 23 Part sample that undergo temperature of (a) 500°C (c) 450°C (d) 400°C with heating rate of 10°C/min and (d) swelling and crack of parts surface under 40°C/min at the temperature of 400°C

Figure 6 - 24 Weight loss of thermal debound part with temperatures of 10 replications each

Figure 6 - 25 Effect of temperature in weight loss of part after thermal debinding process

Figure 6 - 26 XRD pattern of part under 400°C thermal debound

Figure 6 - 27 Peaks of highlighted area from Figure 6-26 shows increment of α- martensitic peak and decrease in γ-austenite besides peak shifted

Figure 6 - 28 XRD pattern of part under 450°C thermal debound
| Figure 6 - 29 | Peaks of highlighted area from Figure 6-28 shows increment of $\alpha$- martensitic peak and decrease in $\gamma$-austenite besides peak shifted | 180 |
| Figure 6 - 30 | XRD pattern of part under 500°C thermal debound | 180 |
| Figure 6 - 31 | Peaks of highlighted area from Figure 6-18 shows increment of $\alpha$- martensitic peak and decrease in $\gamma$-austenite besides peak shifted | 181 |
| Figure 6 - 32 | SEM/EDS of water atomised SS316L | 181 |
| Figure 6 - 33 | SEM of 400°C Thermal debound part morphology and EDS analysis | 182 |
| Figure 6 - 34 | SEM of 450°C Thermal debound part morphology and EDS analysis | 182 |
| Figure 6 - 35 | SEM of 500°C Thermal debound part morphology and EDS analysis | 182 |
| Figure 6 - 36 | Shrinkage value relatives to temperature of debinding process | 183 |
| Figure 6 - 37 | Effect of heating rate with 400°C temperature on percentage of weight loss | 184 |
| Figure 6 - 38 | Surface morphology of cross sectional (a) green part (b) after solvent part (c) brown part (d) after sinter part | 185 |
| Figure 6 - 39 | (a) comparison of part after each process (b) shrinkage value after each MIM process | 186 |
| Figure 6 - 40 | Density of part after each process | 188 |
| Figure 6 - 41 | Density of part after each process in MIM relative to theoretical density | 189 |
| Figure 6 - 42 | Surface morphology of the sintered part (a) after polishing and (b) after etching | 189 |
| Figure 6 - 43 | Surface morphology of sintered part after etching | 190 |
| Figure 6 - 44 | SEM and EDS analysis on the etched sintered sample | 191 |
| Figure 6 - 45 | Section sketch of microhardness test on the sintered part | 191 |
| Figure 6 - 46 | Hardness test for grip’s cross sectional area (A-A’) | 192 |
| Figure 6 - 47 | Hardness test value on the surface of Grip area ‘B’ | 192 |
Figure 6-48  Hardness test value on the surface of neck area ‘C’  193
LIST OF SYMBOLS AND ABBREVIATIONS

FOG - Fats, Oils and Grease
RWL - Restaurants Waste Lipids
PMMA - Poly(methyl methacrylate)
PP - Polypropylene
PE - Polyethylene
LDPE - Low Density Polyethylene
HDPE - High Density Polyethylene
CPVC - Critical Powder Volume Concentration
TGA - Thermal gravimetric Analysis
DSC - Differential Scanning Calorimeter
ASTM - American Society for Testing and Materials
MPIF - Metal Powder Industries Federation
SEM - Scanning Electron Microscope
XRD - X-Ray Diffraction
μMIM - Micro Metal Injection Molding
PIM - Powder Injection Molding
FCC - Face Center Cubic
BCC - Body Center Cubic
<table>
<thead>
<tr>
<th>Acronym</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>PM</td>
<td>Powder Metal</td>
</tr>
<tr>
<td>$D_{10}$</td>
<td>Distribution of powder at 10%</td>
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<tr>
<td>$D_{50}$</td>
<td>Distribution of powder at 50%</td>
</tr>
<tr>
<td>$D_{90}$</td>
<td>Distribution of powder at 90%</td>
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<tr>
<td>CHMA</td>
<td>Cyclohexyl Methacrylate</td>
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<tr>
<td>DMPT</td>
<td>Dimethylpara Toluidine</td>
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<tr>
<td>EVA</td>
<td>Ethylene-vinyl acetate</td>
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<tr>
<td>PEG</td>
<td>Polyethylene Glycol</td>
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<tr>
<td>PW</td>
<td>Paraffin Wax</td>
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<tr>
<td>PK/PS</td>
<td>Palm Kernel/Stearin</td>
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<tr>
<td>MW</td>
<td>Microcrystal paraffin wax</td>
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<tr>
<td>SA</td>
<td>Stearic acid</td>
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<tr>
<td>OA</td>
<td>Oleic acid</td>
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<tr>
<td>HSA</td>
<td>12-hydroxystearic acid</td>
</tr>
<tr>
<td>$V_p$</td>
<td>Volume powder</td>
</tr>
<tr>
<td>$V_L$</td>
<td>Volume liquid</td>
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<tr>
<td>$\eta$</td>
<td>Viscosity, Pa.s</td>
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<tr>
<td>$\dot{\gamma}$</td>
<td>Shear rate, s$^{-1}$</td>
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<tr>
<td>$n$</td>
<td>Flow behavior index</td>
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<tr>
<td>$E_a$</td>
<td>Activation energy</td>
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<tr>
<td>$R$</td>
<td>Gas constant</td>
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<tr>
<td>$T$</td>
<td>Temperature</td>
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<tr>
<td>$B$</td>
<td>Reference viscosity</td>
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<tr>
<td>$\alpha_{STV}$</td>
<td>Mouldability index</td>
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<tr>
<td>DOE</td>
<td>Design of Experiment</td>
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<td>Symbol</td>
<td>Description</td>
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<tr>
<td>$D_e$</td>
<td>Interdiffusion coefficient</td>
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<tr>
<td>$\varnothing$</td>
<td>Removed fraction of soluble binder</td>
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<tr>
<td>$A$</td>
<td>Area</td>
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<td>$t$</td>
<td>time</td>
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<tr>
<td>$N_A$</td>
<td>Avogadro constant</td>
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<tr>
<td>$N_2$</td>
<td>Nitrogen gas</td>
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<tr>
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<td>Hidrogen gas</td>
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<tr>
<td>CIM</td>
<td>Ceramic Injection Moulding</td>
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<tr>
<td>DA</td>
<td>Dissociate Ammonia</td>
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<tr>
<td>Fe</td>
<td>Ferum</td>
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<tr>
<td>Cr</td>
<td>Chromium</td>
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<tr>
<td>Ni</td>
<td>Nickel</td>
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<tr>
<td>$S_W$</td>
<td>Distribution slope parameter</td>
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<tr>
<td>FTIR</td>
<td>Fourier transform infrared spectroscopy</td>
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<tr>
<td>$W_B$</td>
<td>Weight of binder</td>
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<tr>
<td>$W_P$</td>
<td>Weight of powder</td>
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<tr>
<td>$\rho_B$</td>
<td>Density of binder</td>
</tr>
<tr>
<td>$\rho_P$</td>
<td>Pycnometry Density of powder</td>
</tr>
<tr>
<td>$\bar{T}$</td>
<td>Average S/N ratio</td>
</tr>
<tr>
<td>S/N</td>
<td>Signal to Noise</td>
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<tr>
<td>$\bar{y}$</td>
<td>Mean</td>
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<tr>
<td>$\delta_{N-1}$</td>
<td>Standard deviation</td>
</tr>
</tbody>
</table>
# LIST OF APPENDICES

<table>
<thead>
<tr>
<th>APPENDIX</th>
<th>TITLE</th>
<th>PAGE</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Data of binder formulation calculation and rheology</td>
<td>219</td>
</tr>
<tr>
<td>B</td>
<td>Data of stainless steel (SS316L) powder particle size, Critical Powder Volume Concentration (CPVC) and SEM/EDS analysis</td>
<td>230</td>
</tr>
<tr>
<td>C</td>
<td>List of Publications</td>
<td>236</td>
</tr>
<tr>
<td>D</td>
<td>Specification of stainless steel powder (SS316L), Polypropylene, Restaurant waste lipids supplier</td>
<td>238</td>
</tr>
</tbody>
</table>
CHAPTER ONE

INTRODUCTION

Towards implementing the green technology, four pillars are being listed for improvement, which are energy, which is seeking to attain energy independence and promote efficient utilizations. Seconds is to enhance national economic development through the usage of green technology while the third one is more concern on society where to improve the quality of life for all generations. Finally, it is to conserve and minimize the impact of development on environment. A sector, which is promising in the usage of green technology, is identified to be in energy sectors, building, transports and water waste management sectors. Sustainable Development will require major changes with respect to production and consumption patterns in our societies, including materials, energy sources and production processes used in industry, the products and services offered, as well as the organisation and management of supply chains and the governance of firms. Industry, and the manufacturing sector in particular, is therefore one of the key addressees of the Sustainable Development strategy since a more sustainable manufacturing sector can make significant contributions to attain the objectives. This is stated in the Brundtland Report [1] about the key concepts towards Sustainable Development "the idea of limitations imposed by the state of technology and social organization on the environment's ability to meet present and future needs" where here the technology is more towards the manufacturing sectors to implementing the sustainable manufacturing with consuming less materials resources and energy.

With rapid growth of human population, index of pollution results from RWL also will increase. It is found that, the change of habits will also increase the solid waste produce by the human [2]. Urbanisation also lead to change in diets where more
food with higher in fat, animal products, sugar and processed food become the priority [3]. Due to these rapid changes of dietary more pollution is created results from fat, oil and grease (FOG) to the water stream in sewage. This will results in high toxicity of the wastewater [4] due to increasing use of oil and grease in high-demanded oil-processed foods, establishment and expansion of oil mills and refineries worldwide, as well as indiscriminate discharge of oil and grease into the water drains, domestically and industrially. It will also increase the government expenditure due to pipe blockages, pipe break excessive inflows and power failure. Clean up cost could rise thousands of dollars to the municipalities as claimed by Hong Kong Drainage Service Department [5]. The increasing of this source of food tend to increase the production of cooking oil in factory and this is true through its export data of crude palm oil from Malaysia palm Oil Council [6]. Besides the increasing of cooking oils demand, the consumption of animal meat also increase which also contribute to the waste water pollution. Reuse of such wastes as a sustainable construction material appears to be viable solution not only to pollution problem but also to the problem of the land-filling and high cost of building materials [7].

Powder injection moulding (PIM) which consists of metal injection moulding (MIM) and ceramics injection moulding (CIM) is one of the major manufacturing processes used to generate small parts with intricate geometries, thin walls and in large production batches [8], [9]. It combines the flexibility and high productivity of the plastics injection moulding with the powder metallurgy method of sintering. Therefore, it has the capability to manufacture a wide range of components having multifaceted shape, high performance, application areas are various ranging from automotive (locking mechanisms, transmissions synchronisers, and airbag sensors), electronics (computer hard disk drive magnet), defences and aerospace (rocket nozzle guidance system, aircraft engine screw seal), to medical industry (orthodontic brackets, medical forceps) and jewellery (wristwatches). A propose materials, common metals, alloys, ceramics and carbides are frequently encountered [10].

Then, since the sintering of a compacted powder is similar for a part obtained by injection or press moulding, the recipe points in MIM turned out to be how to make the metal flow into the mould and how to retain the shape of the moulded part until it begins the sintering. Dispersing the powdered metal into a binder to form a gloop that flows at high temperature and becomes solid at room temperature commonly solves
this difficulty. As a result, the moulded part retains its shape after injection moulding and may be handled and processed safely.

The research of binder is the heart of this technique in particular, the binders in the feedstock strongly determine MIM quality [11]. The recipe points in MIM binder turned out to be how to make the metal flow into the mould and how to retain the shape of the moulded part until it begins the sintering. Dispersing the powdered metal into a binder to form a gloop that flows at high temperature and becomes solid at room temperature commonly solves this difficulty. They provide adhesion among powdered particles and improve the mechanical properties of feedstock and prevent separation phenomena among binders and powders. As a result, the moulded part retains its shape after injection moulding and may be handled and processed safely. cost reduction and less environmental issues of binder also plays significant role in proper selection of binder besides to achieve low viscosity of the feedstock [11], [12]. Usually binders are mixture of several organic compounds where the main ingredients are waxes and synthetic polymers. The purpose of the polymer is to convey rigidity to the part when cold, while the wax reduces the viscosity and flow ability of the binder and the additives reduce particle severance and segregation [13]. It is usual to convert the powder-binder mix, the so-called feedstock, into solid pellets by a granulation process. These feedstock pellets can be stored and fed into the moulding machine as required.

Parts that are produce from the injection moulding machine are called green parts where this parts contains of metal powder, polymer, wax and additives. This is not a finalised part since it will go another process to get the finalized parts. The green parts will then undergoes another process called debinding process where the primer and secondary binder will be flowed out of the parts through solvent, catalyst wicking or thermal debinding. Sometimes this thermal process will simultaneously prepared with the sintering process since the sintering also required certain temperature in bonding the metal powder in order to get the desired density, strength and shape of final products. The full view of the process is shown in Figure 1-1;
Due in promoting the sustainability development in this manufacturing area, waste materials from the restaurants waste lipids (RWL) or fats, oils and grease (FOG) derivatives from restaurants will be used here since it is believed that it can contribute in some area of metal injection moulding process especially in preparing the binder systems for powder metals feedstock. This abundant resource is increasing with the increasing human population, which make it possible solution in reducing and recycling waste as alternative for current binder in the markets for MIM.

1.1 Problems Statement

The use of RWL in MIM process is a new idea besides it’s used in the biodiesel sector. It is the challenge how this waste can be used in the manufacturing area since it has composition of different organic materials. It is found that this RWL contains high energy and oil recovery which can be obtained from this abundant source of energy [15]. In terms of MIM, process the RWL seems could be used in the production of MIM feedstock since it has the mixture of animal fats, vegetable oils which compose of several organic acids [16].

In MIM, the miscible issues of RWL and polymer will be analysed since the mixing ability of the binder components is very important in providing good
interaction of metal powder and binder for flowability of the feedstock. Results of this analysis could lead to better rheological characteristic of the feedstock.

Since RWL contains high fats and oils recovery which act as a lubricant in feedstock during injection moulding process [17], other issues such as crack, distortion, shot short, density and strength of the injected moulded parts or green parts will be analysed [18]. It is expected that the binder components in MIM should not retain in green parts and being removed before sintering processes. Therefore finding the suitable extraction process of the binder need to be considered since improper removal could results in several defect on the green parts which affect the sintering process and the materials characteristic of the sintered parts [19].

1.2 Objectives

The main objective of the research is to evaluate the application of RWL in metal injection moulding. In order to achieve the objective, several sub-objectives need to be implemented, which are:

a) To investigate the mixing and rheological characterization of different binder formulation between PP and RWL and optimum powder loading for metal injection moulding feedstock.

b) Optimising the injection moulding parameters of metal injection moulding process for highest green density and strength by means of Taguchi method for RWL as binder component.

c) Determine the optimum effect of debinding conditions in removing RWL binder component from the green compact.

d) Determine the mechanical and microstructure analysis of the sintered part under graphite vacuum furnace.
1.3 Scopes

Scopes of this research focused on process ability of using novel binder system in order to produce micro metal parts. In order to obtain this, this research will cover the following scopes:

a) Feedstocks of the µMIM were prepared base on water atomised Stainless Steel powder SS316L with mean size of 6µm, Polypropylene and RWL as a backbone binder and lubrication/surfactant respectively.

b) Mixing homogeneity characteristic was based on mixing time between 45 minutes and 90 minutes. Homogeneity of the feedstock will be monitored base on density and TGA and DSC analysis of the feedstock.

c) Characteristic of feedstock produced from the mixing were analysed on rheological properties base on shear rate, shear stress, viscosity, activation energy, flow index, mouldability index and temperature.

d) Optimization of the metal injection process were done base on Taguchi Method where the parameter will be analysed are injection pressure, injection temperature, mould temperature, injection time and holding time.

e) Optimum solvent debinding was based on type of solvent (hexane and heptane), solvent temperature, and time and diffusion rate. Optimum thermal debinding was optimised base on the effect of heating rate and temperature on oxidation of powder and other defects such as warping, swelling and crack.

f) Mechanical properties and characteristic of the sintered parts under heating rate of 5ºC/min, sintering temperature of 1360ºC, sintering atmosphere under high vacuum furnace and dwell time of 100 minute were investigated besides the carbon contents by means of SEM/EDS and XRD.
1.4 Research methodology

In order to achieve the objectives of this study, the following methodologies were used as a guideline during the course of the study.


b) Determination of degradation and melting temperature of the binder components which are polypropylene (PP) and restaurant waste lipids (RWL) for mixing purposes.

c) Determining the optimum binder formulation and powder loading base on rheological behaviour and characteristic using capillary rheometer Instron CEAST SmartRHEO 10 (ISO 11443, ASTM D3835 and DIN 54811).

d) Optimizing the injection parameters for density and strength of green compact by means of MPIF standard 15 and density of specimens standard ISO 527-2 tensile bar for sintering.

e) After optimising the injection parameter has been done, green parts were allowed to experience the solvent debound or extraction process where optimising in terms of temperature, solvent’s type and time of extraction were done. Effect of weight loss and diffusion coefficient of RWL with respect to temperature, solvent’s type and extraction time were analysed.

f) After solvent process, removing the polymer or backbone binder from the brown compact was done by thermal pyrolysis/debound as suitable heating rate and temperature. Selection of degradation temperature for polymer were based on TGA results and temperature, which produced high weight loss of the brown compact, will be selected without any defect on the brown compacts.

g) Sintering process will be done base on the previous researchers such as heating rate, temperature and dwelling time. Mechanical and microstructure properties of the sintered part was done base on MPIF standard 10/ASTM E8 for tensile strength, MPIF standard 51 for
microhardness properties and ASTM 112-13 for grain size properties of the sintered parts.
CHAPTER TWO

LITERATURE REVIEW

2.1 Powder Injection Moulding

Powder injection moulding (PIM) process is the adoption process of plastic injection moulding process. The difference is that the polymer is filled with disperse metal and ceramic powder and transported into the mould cavity to form the mould cavity shape. It is a process of capable in producing small intricate complex part shapes with combining multiple parts into single one and at medium to high quantity production [10], [20]. It has the ability to handle very fine metals or ceramics powder that can be sintered to high densities near to its bulk metals or ceramics. Acceptable ductility and strength could be produced using this technique. Besides its attractive process, wide range of ceramics and metals could be processed with this technique ranging from low alloy steels, stainless steel, magnetic alloys, nickel alloys, tool steels and titanium alloys which become limitation for die casting process. Fine details such as blind holes, recesses, sharp edges and internal or external threads which becomes limited process for investment casting becomes much easier using PIM [21]. PIM could divided into three categories which are ceramics [22]–[28], metals [17], [29], [30] and cermets [31]–[33].

2.2 Metal Injection Moulding (MIM)

Metal injection moulding is subdivision of powder injection moulding process. The composites fabricated by MIM can be divided into refractory metal based metal matrix composites (MMC), titanium based, intermetallic based and steel based. The MIM direction has enabled the fabrication of MMCs containing ingredient materials that are
not compatible in molten state and difficult to fabricate by conventional routes [21], [34].

MIM relies on shaping metal particles and subsequently sintering those particles. Hence MIM products are competitive with most other metal component fabrication routes, and especially are successful in delivering higher strength compared with die casting, improved tolerances compared with investment or sand casting, and more shape complexity compared with most other forming routes [35].

MIM production involves several processing steps which are mixing of binder and powder to form feedstock, injection of green part, debinding and sintering [36]. Every steps of processing plays significant roles in producing good parts with acceptable mechanical and dimensional properties. Mistakes could not be repaired in subsequent process.

Various metals powder have been used ranging from stainless steel, titanium, high speed steel, Inconel and Nickel Titanium alloy to form a feedstock [33], [37]–[43]. Various powder particle shapes have been used in MIM process ranging from sphere, rounded and flaky shapes. This particle shapes were depend on the types of processing used in producing the metals powder such as air atomising particles which results in sphere shape particles as compared water atomised which results in rounded or irregular shapes as shown in Figure 2-1 [44].

Figure 2 - 1: Production of metal powder by gas atomisation, centrifugal and water atomisation [44]
2.3 Binder

Binder plays a significant role in transporting the metal powder to the mould cavity during injection process. It should not interact chemically with the metal powder during the subsequent process which may alter the composition of the final sintered products. Many types of polymers range from thermoplastic and thermosetting can be used as a backbone binder in MIM which hold the part retention during solvent, wicking or thermal debinding [45]–[48]. Backbone binder itself is unable to produce good flow in injection moldings process, which results in higher viscosity and shear rate appreciated in the range of below 1000 Pa.s for viscosity and $10^2$ to $10^5$ s⁻¹ for shear rate. Therefore waxes in terms of vegetable oils, camphor, naphthalene, dish soap and fish oil are common for lubrication in MIM feedstock to improve the flowability of the feedstock.

Waxes commonly has low melting temperature which is an advantages during debinding process in creating pores for the ease of thermal degradation of backbones binder. With the help of pores after removing these waxes, quickest time possible for removing backbone could be done which also reduced the possible defects on the part in terms of part distortion, swelling and cracks. Waxes also serve as a good wetting agent due to short molecular chain lengths low viscosities and decompose at low temperature with small volume change compares to other polymers.

Fraction between polymer and waxes is roughly equal in its proportions. With the aid of current research, this proportions evolved with just minimum by volume or weight ratio of polymer is enough in holding the powder particles in place. Nowadays, proportions of 20 to 80% by volume between polymer and waxes are possible with appreciation of surfactants such as stearic acids (SA) and oleic acids (OA) [49]–[53]. These surfactants will increase the wettability of the binder constituents on powder particles surface which results in reducing the amount of polymer in MIM feedstock that can contribute in minimizing carburization on the sintered part during sintering which could contributed to carbide formations which results in much brittle part [54]–[56]. It is clear that the binder is the key that provides the rheological properties and determines whether the resulting feedstock can be injection moulded without introducing defects [57].
2.3.1 Backbone binder

Since polymer is the preferable backbone binder in producing acceptable feedstock, many types of polymer have been used. Polymer from thermoplastic gives an advantage among others type of polymer since the ability of recyclable of the feedstock as compare to thermosetting which degrade upon reheating. This is due to the thermosets polymer has the formation of chemical crosslinks by covalent bonds which upon complete polymerization become infusible solids that will not soften when reheated. Thermoplastics comprise essentially linear or lightly branched polymer molecules, while thermosets are substantially crosslinked materials, consisting of an extensive three-dimensional network of covalent chemical bonding [58]. Relative to thermoplastic materials, it can be reheated several time, which likely become favourable in the MIM feedstock. Only several polymers from thermoosetting materials being encountered for MIM feedstock, which is done by Castro et al., [45] where they used cyclohexyl methacrylate (CHMA), ethyleneglycol dimetacrylate (DMEG) and dimethylpara toluidine (DMPT) for making AISI 316L feedstock in analysis of mechanical properties and pitting corrosion resistance being explored although much has been listed by Randall and Bose [59].

Various thermoplastic polymer have been encountered to be backbone polymer for MIM such as polyethylene (HDPE, LDPE, LLDPE, MDPE), polypropylene (PP), polystyrene (PS), ethylene vinyl acetate (EVA), polyethylene glycol (PEG) and polymethyl methacrylate (PMMA) as shown in Figure 2-2 [22], [49], [60]–[63].

![Chemical structures of various polymers](image)

Figure 2 - 2: Examples of polymers used in different binder formulations [62].
The selection of polymer usually base on the type of application. Although polymer as binder in MIM should not dictate the final composition of the molded materials, selection of the types of polymer also plays critical role in producing good mechanical properties of the sintered materials due to residue produced from it through thermal debinding. In some extent polymer selection need to consider environmental issues due to its degradation has potential effects on human health such as styrene monomers used in acrylonitrile-butadiene-styrene (ABS), and styrene-acrylonitrile resins. Styrene is classed by International Agency for the Research on Cancer (IARC) as possibly carcinogenic to humans[64]. Vinyl Chloride is used essentially to produce polyvinyl chloride (PVC) resins also has some hazards potential such as halogen acids[65].

Degrading these polymer during thermal debinding process could introduce residual carbon on the sintered parts. Therefore such analysis in carbon contents have become the interesting topic to be discussed. Carbon content after thermal debinding was much influenced the degradation behaviour of the polymer, interaction of polymer with the powder particles, chemistry of powder surfaces, thermal debinding atmosphere and oxidation temperature of the powder [31].

2.3.1.1 Ethylene Vinyl Acetate (EVA)

EVA has low melting temperature which is approximately 86°C with degradation temperature of 520°C [63]. It has some of the properties of a low density polyethylene but increased gloss (useful for film) and being used as components of binder in MIM because of its properties of non-toxic materials. It has been used widely ranging from titanium [33], [36], [66], [67], stainless steel [12], [68]–[70], W–Cu alloys [71], Inconel 718 Alloy [72]. It was used by Demers et al., [63] in analysing the segregation measurement of powder injection molding feedstock using thermogravimetric analysis, pycnometer density and differential scanning calorimetry techniques. Low pressure MIM is possible with EVA as backbone binder since it has low melting temperature. Fan et al., [49] used EVA in determining the effect of surfactant addition on rheological behaviours of ultrafine 98W-1Ni-1Fe suspension feedstock and results in better rheological properties with additional of surfactant. Youhua et al., [72] used
EVA with other components of binder in preparing the Inconel718 Alloy feedstock to be used for MIM process in investigating the effects of sintering processes, hot isostatic pressing and heat-treatment on the density, microstructure and mechanical properties of the alloy for finding a proper way to prepare a high performance nickel based alloy through MIM-sintering-HIP-heat treatment technology. Highest sintered relative density of 98% can be achieved with superior mechanical properties was achieved. Li et al., [73] used EVA in preparing the 17-4PH stainless steel for feedstock preparation where low pressure injection moulding process was possible with the used of paraffin wax (PW) and stearic acids. The lowest flow exponent for 68% powder loading indicates that there is a best powder binder ratio for MIM feedstock to get quick powder repacking and binder molecule orientation during moulding.

2.3.1.2 Polyethylene (PE)

Polyethylene polymer in MIM can be devided into low density polyethylene (LDPE), high density polyethylene (HDPE) and medium density polyethylene (MDPE). It is most common plastic used for packaging, toys and bullet proof vest. It consists of nonpolar, saturated, high molecular weight hydrocarbons. In MIM, PE has been used for binder in producing feedstock such as titanium feedstock [74]–[77], stainless steel [52], [78], [79], zirconia [26], [80], cemented tungsten carbide [81] and among other materials. The used of this binder shows good flowability of feedstock inside the mould cavity and rheological characteristic. Subsequent process of the MIM showing good part retention after solvent debinding without any parts defect such as swelling or part cracks. The sintering condition with this type of binder shows good mechanical properties.

2.3.1.3 Polymethyl Methacrylate (PMMA)

Using PMMA as binder in MIM was found in making the feedstock of stainless steel [82], [83], titanium [84], tungsten carbide cobalt [85] and many other material applications. The wide used of PMMA as backbone binder was due to its ability to mix of powder with PMMA at room temperature with the help of acetone. Good part shape retention also contributes to this wide usage of this polymer in MIM. Good
rheological characteristics and high powder loading on water atomised 316L powder was determined by Ibrahim et al., [86]. Subsequent process of solvent, thermal debinding and sintering also shows good mechanical properties and characteristic. Table below shows application of PMMA as binder by other researchers in their research.

2.3.1.4 Polypropylene (PP)

PP also known as polypropene, is a thermoplastic polymer used in a wide variety of applications including packaging and labeling, textiles, stationery, plastic parts and reusable containers of various types, laboratory equipment, loudspeakers, automotive components, and polymer banknotes. An addition polymer made from the monomer propylene, it is rugged and unusually resistant to many chemical solvents, bases and acids. Kong et al., [37] did comprehensive study of determining the best binder formulation between LDPE and PP as backbone binder. The PP and LDPE was mixed with secondary binder of carnauba wax (CW) and paraffin wax (PW) with addition of stearic acids (SA) as wetting and lubrication agents. Different molecular weight of PP and LDPE have been tested with 60% powder loading of gas atomised 316L stainless steel powder (D$_{50}$ = 3µm). The best selection of binder were based on mixing torque and rheological behaviour of the feedstock with different binder composition. They found that, binder composition containing the PP with PW and SA was better since it produced low mixing torque and low viscosity for rheological behaviour. This binder formulation was then being further analysed for searching the critical and optimum powder loading of stainless steel powder. From their results a good sintered parts was obtained and optimal powder loading of 64 vol.% was achieved. Other researchers also used PP as backbone binder in making their feedstock for stainless steel powder and found that optimum powder loading were obtained besides good rheological characteristics and sintered parts [87][88]. Other than that PP was able to be mixed with other secondary binder like PEG, PW, palm kernel, palm stearin and CW [10], [51].

Besides its usage in making the MIM feedstock, PP also being used in CIM as binder in making the CIM feedstock. Onbattuveli et al., [89] used PP as backbone binder determining the effects of nanoparticle addition on binder removal of silicon
carbide injection moulded specimens. No defects were found for their debinding analysis with the usage of PP as backbone binder. Aggarwal et al., [90] used PP in their niobium feedstock for determining the master decomposition curve for binder used in PIM where this method can predict the remaining amount of binder during the debinding process, and such can help to optimize the binder composition without additional experiments.

In making a feedstock of titanium, PP also has been used as backbone binder by other researchers [91],[74] and found that thermal debound and sintered of micro parts shows no sign of defects. This indicates that the usage of PP as backbone binder can be used as binder even for the difficult or critical powders such as titanium feedstock.

2.3.2 Secondary binder

Secondary binder was name because of its ability to decrease the viscosity and increase the wettability and miscibility of the feedstock during the injection moulding process. It was not meant for powder particle holder since it has low molecular weight which results in low melting temperature. Many types of polymer, waxes and plasticizers has been encountered as secondary binder in MIM and CIM such as polyethylene glycol (PEG), carnauba wax [92], paraffin wax [70], peanut oil, palm kernel and palm stearin.

2.3.2.1 Polyethylene Glycol (PEG)

PEG is a polyether compound with many applications from industrial manufacturing to medicine. Due to its low melting temperature, PEG was used as secondary binder in most MIM application related to feedstock. Unlike the polymer/wax binder systems which used organic solvents frequently in solvent debinding process which are flammable, carcinogenic and environmentally unacceptable, PEG is soluble in water. This advantages contribute to very safe chemicals and are used quite extensively in food industry and allowed from the local water authorities to dump the water/PEG containing solvents into the drain after debinding [57], [93]. Yang et al., [57] use different molecular weight of PEG in determining its effect on rheological behaviour of injection moulding alumina feedstock. They found that PEG with highest molecular
weight (20K) was better as compared to low molecular weight (1K and 1.5K). The backbone binder they used for their research was polyethylene wax. Chen et al., [77] used PEG along with PMMA as backbone binder and stearic acid for wettability agents. Analysis of PEG removal inside the water was increase with temperature and diffusion of PEG was low at room temperature. Porosity increase with debinding time base on the surface porosity of the green parts. This shows that PEG usage in MIM as binder also shows good flowability in MIM. Krauss et al., [93] develop a model for PEG removal in alumina injection moulded part. They found that the pore diameter remain the same throughout the debinding time but the volume or number of pores inside the specimens was increased. Hayat et al., [84] use the same experimental method by Yang et al., [57] where the same molecular weight of PEG was used. They conclude that the PEG with higher molecular weight shows good rheological behaviour due to the interaction between powder and polymeric binder, as a result of increased number of hydrogen bonds on the longer PEG molecule chains.

The used PEG with cellulose acetate butyrate (CAB) [84], [94], [95] as backbone binder results in pseudoplastic behaviour and a global viscosity model involving all of the dependent variables, including shear rate, temperature, solid loading and particle sizes was achieved. Sharmin and Schoegl [23] used PEG (6000 molecular weight) along with EVA in analysing the two-step debinding and co-extrusion of ceramic-filled polyethylene butyl acrylate (PEBA) and EVA blends. Good rheological behaviour was achieved. Results of solvent debinding of EVA-PEG was better as compare to PEBA-PEG binder due to compatibilities between PEG and the thermoplastic binder which in this case EVA shows better compatibility with PEG than PEBA, and the slower removal rate and higher torque is attributed to a finer dispersion of PEG within the blend. Thavanayagam et al., used PEG of 8000 molecular weight as binder in titanium feedstock and good rheological behaviour was achieved.

2.3.2.2 Paraffin wax (PW)

Paraffin wax is a white or colourless soft solid derivable from petroleum, coal or oil shale, which consists of a mixture of hydrocarbon molecules containing between twenty and forty carbon atoms. Common applications for paraffin wax include lubrication, electrical insulation, and candles.
Detail used of PW was elaborated by Zaky et al., [96] where they use different molecular weight of PW ranging from 378 to 572 to investigate the influence of paraffin wax characteristics on the formulation of wax-based binders and their debinding from green moulded parts using two comparative techniques. The mixing time and temperature were fixed at 150ºC and 30 minutes mixing time. Results of their studies indicates that mixtures of EVA as backbone binder and PW of high molecular weight was better since it has good compatibility and rheological properties as it has viscosity below 10Pas which is suitable to fabricate homogenous feedstock during compression or injection moulding process. They also conclude that solvent immersion is a preferable technique as it saves the amount of solvent used as compared with the evaporation–condensation technique.

You et al., [97] use PW as backbone binder in making low pressure injection moulding in making micro gear. They used nano powder in making the feedstock and successfully producing moulded gear which had a sound surface, uniform shape and homogeneous microstructure even after thermal debinding process. The debound gear of the micro-nano powder underwent isotropic shrinkage and near full densification during sintering. Other application of PW as binder were shown by Sotomayor et al., [98] where they used PW with high density polyethylene (HDPE) as backbone binder with formulation of 50/50 between secondary and primary binder for fremixed of ferritic and austenitic stainless steel. Results shows that the PW was successfully mixed with HDPE and solvent, thermal debinding and sintering process was successfully being done without any defects. Four composition of binder was used by Li et al., [99] in producing the molybdenum feedstock where PW as the major components of the binder beside the HDPE, EVA and stearic acids. These types of binder composition usually used for thermal debinding process only where solvent debinding were not been used. All the injected parts were free from defects.

2.3.2.3 Carnauba wax

Carnauba wax is a wax from a plants which is obtained from the leaves of the carnauba palm by collecting and drying them, beating them to loosen the wax, then refining and bleaching the wax. Carnauba wax contains fatty acid which known to be good agent for binder in MIM [100]. In MIM and CIM, carnauba wax has long been known for
its suitability as binder components. It has melting temperature of 84°C which was higher as compare to other types of waxes [92]. Supriadi et al., [12] used carnauba wax for their wax binder systems and good mouldability shape was produced due to its low heat capacity which in turns has high cooling rate and producing good shape retention. Ahn et al., [101] has include carnauba wax in their analysis of determining the effect of powders and binders on material properties and molding parameters in iron and stainless steel powder injection molding process. All the properties of the binder composition shows good pseudoplastic behaviour.

2.3.2.4 Palm kernel and stearin

Palm kernel and stearin was comes from palm oil plants which contains rich amounts of fatty acids. The composition of fatty acids from palm kernel is shown in Table 2-1;

Table 2 - 1: Fatty acid composition (%) of NIE, EIE and CIE 50:50 PO:PKO blend fractions [102]

<table>
<thead>
<tr>
<th>Fatty acid</th>
<th>Fractions</th>
<th>NIE 50:50 PO:PKO</th>
<th>EIE 50:50 PO:PKO</th>
<th>CIE 50:50 PO:PKO</th>
</tr>
</thead>
<tbody>
<tr>
<td>C12:0</td>
<td>Olein</td>
<td>23.45</td>
<td>31.27</td>
<td>23.71</td>
</tr>
<tr>
<td>C14:0</td>
<td></td>
<td>6.46</td>
<td>4.89</td>
<td>8.50</td>
</tr>
<tr>
<td>C16:0</td>
<td></td>
<td>28.49</td>
<td>10.15</td>
<td>25.16</td>
</tr>
<tr>
<td>C18:0</td>
<td></td>
<td>3.27</td>
<td>4.23</td>
<td>2.89</td>
</tr>
<tr>
<td>C18:1</td>
<td></td>
<td>31.45</td>
<td>40.58</td>
<td>32.37</td>
</tr>
<tr>
<td>C18:2</td>
<td></td>
<td>6.67</td>
<td>8.98</td>
<td>7.37</td>
</tr>
</tbody>
</table>

As can be seen C18:0 and C18:1 were the carbon number of stearic acid and oleic acid. This shows that this palm kernel were suitable to be used to replace the function of wax and surfactant in a binder system to ensure good wetting of the powder [51]. Omar et al., [51], [79], [103] quite intensively used palm kernel or stearin in their research of producing 316L stainless steel powder feedstock. With this binder composition, solvent and thermal debinding was successfully being done without any
defects. Sintering process of the parts also indicates good achievement where relative density of 97% was achieved.

2.3.3 Surfactants

Polymeric surfactants are essential materials for preparation of many disperse systems, of which we mention dyestuffs, paper coatings, inks, agrochemicals, pharmaceuticals, personal care products, ceramics and detergents [50]. In PIM surfactants was used to avoid particles agglomeration, wetting agents and lubrication. Surfactants such as oleic and stearic acids was used to improve powder dispersion during mixing [104]. Several analysis has been done in determining the influence of surfactant on suspension structure and green microstructure of injection-moulded parts.

Fraction between polymer and waxes is roughly equal in its proportions. With the aid of current research, this proportions evolved with just minimum by volume or weight ratio of polymer is enough in holding the powder particles in place. Nowadays, proportions of 20 to 80% by volume between polymer and waxes are possible with appreciation of surfactants such as stearic acids (SA) and oleic acids (OA). The aims to reduce the amount of polymer in MIM feedstock can contribute in minimizing carburization on the sintered part during sintering which could contributed to carbide formations which results in much brittle part. Stearic acids is known for low cost polar molecule with molecular structure of \( \text{CH}_3(\text{CH}_2)_{16} \text{COOH} \) and low melting temperature. This wetting agent will improve wettability of the polymer and powder particles by lowering the contact angle by decreasing the surface energy of the binder-powder interface[59]. The ability of bridging the property of polymer could increase the powder loading and results in lowering shrinkage value and reduces the parts from slumping during thermal debinding and sintering. Surfactants also reacts as internal lubricant (incorporated into the host resin during production or compounding), which promotes fusion, and reduce melt viscosity and friction or increase slipperiness between polymer particles before melting.

Tseng et al., [104] has tested several quantity of stearic acids on the zirconia feedstock. The range of addition of stearic acid was between 3 vol.% to 17 vol.% and was found that the pore size of the reduces with increasing the fraction of stearic acids. This indicates good packing structure of the green parts and also leads to low viscosity
of the feedstock. They also found that although higher contents of stearic acid leads to good viscosity and particles packing, too high fraction of stearic acid also leads to high cracks during thermal debinding.

Comparison between oleic, stearic and 12-hydroxystearic acids on rheological behaviour of alumina powders also been done by Tseng [104]. They conclude that the viscosity of the suspension was lower for stearic acids and followed by oleic and 12-hydroxystearic acids. Li et al., [105] did the analysis of different fraction of stearic acids on making the 17-4PH stainless steel feedstock and found that 0.2% theoretical calculation was enough in producing single molecule layer of stearic acid on the powder surface but higher fraction were needed for irregular shape particles. They also found that as the fraction of stearic acid increase, the wetting angle decrease. Fan et al., [49] have determining the influence of stearic acid during ball milled the ultrafine 98W-1Ni-1Fe and found that it improves the particle sizes distribution of the ball milled powder, reduce the mixing time for homogeneity of feedstock, better rheology at lower temperature but opposite when higher temperature were used and reduces the temperature required for injection moulding of 98W-1Ni-1Fe powder. Therefore, the used of surfactant especially stearic acid for binder in MIM becomes common such as in making the water atomised 316L stainless steel feedstock [52], [106].

2.4 Restaurant waste lipids as binder

Domestic and commercial food establishments generate large volumes of wastewater, in the form of grey water or sullage that contains significant amounts of fats, oil and grease (FOG) or RWL. FOG must be separated from wastewater prior to entering the sewage system, primarily because of its propensity to block municipal sewer lines and disrupt the effective operation of downstream treatment processes [107]. RWL can be recovered efficiently from grease interceptors for biodiesel production or other cosmetics products. RWL is susceptible to hydrolysis because of its inherent high moisture content and the presence of lipases associated with food residuals in the grease interceptors. Since the evolved of engine diesel by Rudolf Diesel which tested his engine with vegetable oils as fuel, many are drawback to see vegetable and animal fats as biodiesel fuel for substituting the fossil fuel in minimizing the environmental
pollutions and reducing the dependence on fossil fuels [108]. The number of waste oils and fats is expected to increase with urbanization, life style changes and nutrition transition due to occupational which time consuming which also shifted the diets towards fast foods [3].

Williams et al., [109] have characterized the FOG deposits in sewers on several locations such as pumping station, sewers and sewage works. They found that percentage of free fatty acids (FFA) were shown as Table 2-3 and Figure 2-3. They also found that the majority of the deposits contain FFA of palmitic, oleic, stearic and linoleic acid.

<table>
<thead>
<tr>
<th>Common name (cis)</th>
<th>Lipid no.</th>
<th>Melting point, °C</th>
<th>% Composition by fatty acid</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Sunflower</td>
</tr>
<tr>
<td>Myristic</td>
<td>C14</td>
<td>55</td>
<td>0</td>
</tr>
<tr>
<td>Palmitic</td>
<td>C16</td>
<td>63</td>
<td>6</td>
</tr>
<tr>
<td>Stearic</td>
<td>C18</td>
<td>69</td>
<td>3</td>
</tr>
<tr>
<td>Oleic</td>
<td>C18:1</td>
<td>13</td>
<td>17</td>
</tr>
<tr>
<td>Linoleic</td>
<td>C18:2</td>
<td>–15</td>
<td>74</td>
</tr>
<tr>
<td>α-Linolenic</td>
<td>C18:3</td>
<td>–11</td>
<td>0</td>
</tr>
<tr>
<td>Arachidic</td>
<td>C20</td>
<td>76</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 2 - 2: % mass fatty acids profiles of cooking fats and oils [109]

Figure 2 - 3: Mean concentrations of fatty acids in the FOG deposits [109]

It is stated that RWL in restaurants sewers line are a major problem and can cause sewer overflows, resulting in environmental damage and health risks [109]. It is the challenge how this waste can be used in the manufacturing area since it has composition of different organic materials. It is found that this RWL contains high energy and oil recovery which can be obtained from this abundant source of energy [15]. In terms of Metal Injection Moulding process the RWL seems could be used in
the production of feedstock since it has the mixture of animal, vegetable fats and several organic acids. Table 2-2 shows the reported fatty acids present in restaurant waste lipids.

Table 2 - 3: Reported fatty acids present in grease trap [110]

Montefrio et al., [107] also found the same FFA in their analysis of fats, oil and grease from grease interceptors for biodiesel production. They found that higher FFA in the grease interceptors and the results were compared with FFA contains in palm oils, animal fats (tallow) and lards as shown in Table 2-4;

Table 2 - 4: Fatty acid and FFA profile of FOG samples and neat palm oil [107]

Since RWL contains of animal fats and vegetable oils, Banković-Ilić et al., [111] done some classification of FFA on several types of animal fats and vegetable
oil as shown in Table 2-5. All the vegetable oils and animal fats contains stearic acids and oleic acid which useful as surfactant for dispersing metal powder in MIM feedstock.

Table 2 - 5: Fatty acids composition of some vegetable oils and animal fats [111]

<table>
<thead>
<tr>
<th>Feedstock</th>
<th>12:0</th>
<th>14:0</th>
<th>16:0</th>
<th>18:1</th>
<th>18:2</th>
<th>18:0</th>
<th>18:1</th>
<th>18:2</th>
<th>18:3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rape seed oil</td>
<td>2.4</td>
<td>4.0</td>
<td>64.4</td>
<td>22.3</td>
<td>8.23</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sunflower oil</td>
<td>6.08</td>
<td>3.26</td>
<td>16.39</td>
<td>73.78</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Soybean oil</td>
<td>10.58</td>
<td>4.76</td>
<td>22.52</td>
<td>52.94</td>
<td>739</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chicken fat</td>
<td>0.5</td>
<td>24</td>
<td>5.8</td>
<td>5.8</td>
<td>58.2</td>
<td>23.8</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Duck tallow</td>
<td>5.1</td>
<td>17</td>
<td>4</td>
<td>59.4</td>
<td>19.6</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Munson Fat</td>
<td>0.2</td>
<td>3</td>
<td>2</td>
<td>241</td>
<td>40.7</td>
<td>2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lard</td>
<td>1.7</td>
<td>23.2</td>
<td>2.7</td>
<td>104</td>
<td>42.8</td>
<td>19.1</td>
<td>647</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Yellow grease</td>
<td>2.43</td>
<td>23.24</td>
<td>3.79</td>
<td>12.96</td>
<td>44.32</td>
<td>657</td>
<td>0.67</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Brown grease</td>
<td>1.65</td>
<td>22.83</td>
<td>3.13</td>
<td>12.54</td>
<td>42.38</td>
<td>12.09</td>
<td>0.82</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Although much discussion on FFA contain in RWL, much was concerning its application on biodiesel [16], [110], [112], [113]. Therefore the use of RWL for MIM binder seems suitable to replace the function of wax and surfactant in a binder system to ensure good wetting of the powder since the RWL were either soft or waxy in room temperature.

2.5 Stainless steel 316L feedstock

Stainless steel powder can be divided into of water atomised and gas atomised powder particles. Water and gas is named based on its process for producing the powder particles. Water atomised have round or irregular shapes as compared to gas atomised which have the spherical shapes. Examples of water and gas atomised 316L stainless steel powder is shown in Figure 2-4.
REFERENCES


