# PA12 NYLON DUST REDUCTION OF SELECTIVE LASER SINTERING PRE-PROCESSING BY OPTIMIZATION OF REFRESH RATE AND POWDER HANDLING

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A dissertation submitted in fulfilment of the requirement for the award of the Doctor Philosophy of Mechanical Engineering

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This thesis is dedicated to my parent, who taught me that the best kind of knowledge to have is learned for its own sake. It is also dedicated to my wife, and children who taught me that even the largest task can be accomplished if it is done one step at a time

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"Thank you for your patience and support"

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#### ABSTRACT

Despite advantages of selective laser sintering (SLS) to print complex and highvolume products, major concern in SLS process are exposures during handling especially during pre-processing. This study aims to formulate percentage of refresh rate with powder handling settings with the introduction of enclosure during preprocessing stages to reduces dust exposures using response surface methodology (RSM) of central composites design (CCD) techniques. This research divides into three phases, as the first phase involves a pilot study to select pre-processing as the main contribution of emission. Analysis of variance and 95% confidence interval were used to identify factors and responses that significantly contributed to the IAQ in SLS workplaces involving 56 set of screening experiments using factorial design. Next, second phase involves series of experiments to formulate refresh rate, collecting powder from mixing machines and transferring activities due to enclosures settings using RSM techniques. Finally, the third phase involves assessing gravimetric based on personal sampling (based on NIOSH 0500 and 0600) for using enhanced model suggested by the RSM. Through screening analysis, it was obtained that refresh rate give significant percentage of contribution (72.73%) to the emission followed by collecting powder from mixing machine and transferring activity. From CCD analysis with 0.816 desirability suggested 100% refresh rate, 32.8% enclosure for collecting powder from mixing machine, and full enclosure for transferring process to reduce PM2.5, PM10, UFP and TSP. The confirmation experiment was also conducted to verify the prediction result using percentage of absolute error (%) with 4.33,3.57, 4.56 and 2.38 for PM2.5, PM10, UFP and TSP. Based on the enhanced model from RSM, performance experiments show acceptable percentage of reduction for 40.6 and 28.8 % of reduction for NIOSH 0500, and 22.7 and 27.3% for NIOSH 0600, respectively. The mathematical model from RSM shows promising strategy in term of control measures in protecting operators in SLS manufacturing industry from occupational exposures.



#### ABSTRAK

Walaupun "selective laser sintering" (SLS) mempunyai kelebihan dalam mencetak produk yang kompleks dan banyak, tumpuan utama ialah serakan habuk ketika praproses. Kajian ini bertujuan meramal peratusan kadar serbuk penyegaran dan tetapan pengendalian dengan penggunaan kepungan ketika pra-proses untuk mengurangkan dedahan habuk dengan menggunakan teknik kaedah respon permukaan (RSM) iaitu reka bentuk komposit tengah (CCD). Penyelidikan ini terbahagi kepada tiga fasa iaitu fasa pertama ialah kajian rintis bagi pemilihan pra-proses sebagai pelepasan utama. Analisis varians dan selang keyakinan 95% digunakan untuk menentukan faktor dan respon yang signifikan kepada kualiti udara dalaman (IAQ) di dalam ruang kerja SLS melibatkan 56 set eksperimen dengan menggunakan reka bentuk faktorial. Fasa kedua ialah eksperimen untuk meramal peratusan serbuk penyegaran, pengumpulan serbuk dari mesin pencampuran dan aktiviti pemindahan oleh tetapan kepungan menggunakan teknik RSM. Fasa ketiga ialah penilaian gravitian untuk model yang telah dipertingkatkan menggunakan cara persendirian (NIOSH 0500 dan 0600). Dengan analisis saringan, didapati serbuk penyegaran memberi kadar peratusan pelepasan yang signifikan dengan 72.73%, diikuti pengumpulan serbuk dari mesin pencampuran dan aktiviti pemindahan. Dengan peratusan kehendak 0.816, habuk saiz PM2.5,PM10, UFP dan TSP, dapat dikurangkan dengan menggunakan 100% serbuk penyegaran, 32.8% kepungan ketika pengumpulan dari mesin pencampuran dan kepungan penuh ketika proses pemindahan. Ekperimen pengesahan di jalankan untuk menilai hasil ramalan dengan menggunakan kadar peratusan kesilapan mutlak (%) dengan 4.33, 3.57, 4.56 dan 2.38 untuk PM2.5, PM10, UFP dan TSP. Dengan model yang dipertingkatkan oleh RSM, eksperimen penilaian prestasi menunjukkan kadar pengurangan habuk yang diterima iaitu 40.6 dan 28.8% untuk NIOSH 0500, dan 22.7 dan 27.3% untuk NIOSH 0600. Model matematik dari RSM ini boleh menunjukkan strategi yang baik dalam mengawal ukur bagi melindungi pengendali SLS di industri dari pendedahan pekerjaan ketika menggunakan serbuk penyegaran.



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## LIST OF SYMBOLS AND ABBREVIATIONS

$CO_2$	-	Carbon dioxide
СО	-	Carbon monoxide
<i>O</i> <sub>3</sub>	-	Ozone
$SO_2$	-	Sulphur oxide
$^{\circ}C$	-	Degree Celsius
g	-	Gram
kg	-	Kilogram
t	-	Time (min)
%	-	%
Т	-	Time (min) % Temperature milligram per metre cubic
mg/m3	-	milligram per metre cubic
m/s	-	metre per second
ppm	-	part per million
cfu/m3		colony forming units per cubic metre
μg/m3	<u>p</u> U-	micron gram per cubic metre
pt/cc	-	particles per cubic centimetre
h	-	hour
т	-	metre
μm	-	micron metre
nm	-	nano metre
AM	-	Additive manufacturing
PBF	-	Powder bed fusion
AM	-	Additive manufacturing
IR4.0	-	Industrial revolution 4.0
FDM	-	Fused deposition modelling
CAD	-	Computer assisted design
3D	-	Three dimensional

Standard tessellation language
Indoor air quality
Indoor air pollution
Selective laser sintering
Stereolithography
Selective laser melting
Polylactic acid
Acrylonitrile Butadiene Styrene
Polyamide nylon 12
Design of Experiment
Response surface methodology
Central composites design
Scanning electron microscopy
Particle size analysis
Department if Occupational Safety and Health
Department if Occupational Safety and Health World Health Organization
National Institute Occupational Safety and Health
Social Security Organization
American Society of Heating, Refrigerating and Air Conditioning
Engineers
American Society for Testing and Materials
Environmental Protection Agency
Health and Safety Executive
Health and Safety Executive
United States

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stl.

IAQ

IAP

SLS

SLA

SLM

PLA

ABS

PA12

DoE

RSM

CCD

SEM

PSA

DOSH

WHO

NIOSH

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SOCSO	-	Social Security Organization
ASHRAE	-	American Society of Heating, Refrigerating and Air Conditioni
		Engineers
ASTM	-19	American Society for Testing and Materials
EPA	<u>P</u> U	Environmental Protection Agency
HSE	-	Health and Safety Executive
HSE	-	Health and Safety Executive
US	-	United States
UK	-	United Kingdom
USECHH	-	Use and Standard Exposure Chemical Hazardous to Health
ACGIH	-	American Conference of Governmental Industrial Hygienists
AAS	-	Active air sampling
PAS	-	Passive air sampling
HVAC	-	Heating, ventilation, and air conditioning
TTE	-	Total time exposure
STEL	-	Short term exposure limit

TWA	-	Time weighted average
OEL	-	Occupational exposure limit
PPE	-	Personal protective equipment
RH	-	Relative humidity
VOC	-	Volatile organic compound
TVOC	-	Total volatile organic compound
PM	-	Particulate matter
PM 2.5	-	Particulate matter size 2.5 µm
PM 10	-	Particulate matter size 10 µm
UFP	-	Ultrafine particle
TSP	-	Total suspended particle
Avg	-	Average
Min	-	Minimum
Max	-	Maximum
Std.Dev	-	Standard deviation
SBS	-	Sick building syndrome
LLVM	-	Standard deviation Sick building syndrome Linear ventilation model
ANN	-	Artificial neural network
CB	-	Control banding
LEV	-	Local exhaust ventilation
ANOVA	-19	Analysis of variance
FCCD	<u>P</u> O '	Face centred central composites design
CCRD	-	Central composite rotatable desing
POAE	-	percentage of absolute error

ICOP Industry code of practice \_

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### **CHAPTER 1**

### **INTRODUCTION**

#### 1.1 Overview

The research background will be thoroughly discussed in this chapter. Then, the issue related to indoor air quality and exposure from additive manufacturing (AM) process and health effect is discussed. Subsequently, in the problem statements, objectives, scopes, and significance of the study, the main issue of IAQ related to the powder bed fusion (PBF) type of AM, specifically for selective laser sintering (SLS), exposure from the powder PA12 feedstock, and development of response surface modelling (RSM) is discussed.



### 1.2 Background of Research

Over the last few decades, the manufacturing industry has been ambitious to pursue high-demand, diverse, and complicated items. The rapid advancement of technology has shifted from subtractive to additive manufacturing due to industrialisation [1]. In 2018, Malaysia launched Industry 4WRD: National Policy on Industry Revolution 4.0 (IR4.0) and put AM as one pillar among the other nine pillars to drive Malaysia forward [2]. The technology of AM offers an alternative to fill the void of conventional manufacturing, making it cost and time efficient. The ideas of AM is to help engineers to realise what they have in mind from design [3]. AM have been used widely in automotive, aerospace, biomedical, energy, consumer goods and others. The process

of AM is the process of building an object or prototype by joining material layer by layer at a time [4].

Unlike the other subtractive processing method, the material block is subtracted by milling, grinding, or drilling. Meanwhile, contrary to traditional manufacturing technologies such as machining and stamping, which manufacture things by removing components from enormous stock or sheet metal, AM generates the final shape by merging layer by layer material. As a result, it can efficiently use raw materials and produce minimal waste while reaching satisfactory geometric accuracy [5]. As a result, when a business uses AM to build products, it can save 50% of the time required to produce products.

There are several types of AM technology presented worldwide. The most commonly used AM technology is the fused deposition modelling type (FDM) which used filament for the material. Despite the popularity of FDM, the powder bed fusion (PBF) type stand out as AM technology to meet the demand of printing complex design and geometries. According to Ngo et al. [6], the key advantages of SLS are its satisfactory resolution and excellent printing quality, making it appropriate for complicated structures. SLS is frequently employed in high-tech applications such as tissue engineering, aircraft, electronics and automobiles. SLS divided into several categories, and the difference between SLS technology is sintering technology and material/powder used as feedstock. SLS uses nylon powder and is suitable for producing parts and prototypes from small to medium-sized complex geometries [7].



SLS uses powder as a feedstock for the production of prototypes and end products. This technology used carbon dioxide (CO<sub>2</sub>) laser beams that heated to the near melting point of the powder, and the laser was sintered and bound to the powder layer by layer. This process will be replicated layer by layer until the component is fully processed. SLS could use a range of materials such as plastics, nylon, metal, polymers and ceramics. The significant benefits of SLS were that the powder could be recycled and used back for the next printing [8].

Based on Figure 1.1, building products using SLS technology starts from the CAD model or design stage. First, the CAD model is converted to stl. format and transferred to the SLS machine. Pre-processing stages start when activity such as weighing, pouring, mixing and transferring process is the process to prepare powder before printing or sintering. After printing complete, post-processing includes

housekeeping, powder cake breakage are done. The un-sintered powder then goes to the powder recycling and storage process.

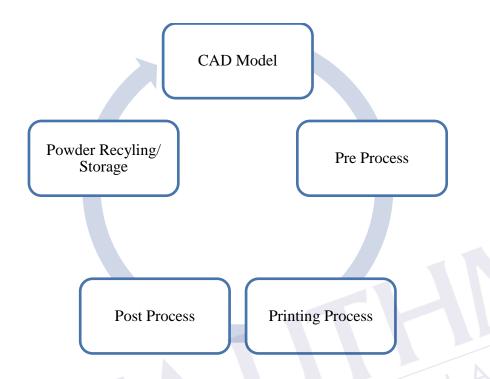


Figure 1.1: General SLS process [9].



During the SLS process, pre-processing and post-processing are done manually by workers [10], [11]. The powdered material heated before drawing each of the slices using a CO<sub>2</sub> laser controlled by a scanning system, selectively fusing the powder particles to produce a solid part. The residual volume from the binding envelope was then either loosen or left un-sintered. The remaining volume of the binding envelope is loose or un-sintered powder. The PA12-based powders decay due to the SLS processing conditions (heat, building time, cooling time, etc.) and must be refreshed or mixed with new material for subsequent use, known as the refresh rate. The sintered pieces may have a rough surface (orange peel) and poor quality if the percentage of fresh powder is insufficient. In addition, the utilisation of virgin powder could increase operating and manufacturing cost. Therefore, operators and manufacturers usually will mix virgin and recycle powder for SLS printing. In contrast, its recycling and refresh rate (mixing powder) could be harmful to the operators that are handling the powders [12], [13].

Due to powders handling during the SLS process, there is possible high indoor concentration and exposure at the SLS workplace, especially during pre-processing and post-processing activities [14]. Therefore, indoor air quality (IAQ) will result from exposure to the SLS process. Thus, SLS operators will affect by SLS operation, which reflects their health and productivity. Although SLS could significantly benefit the industrial application, the concerns regarding the potential impact of the SLS process and activities have been the primary concern recently. Since SLS demand is high, indoor air exposure is highly reflected in the working environment and working practices [15]. Figure 1.2 shows a radar chart adopted from Tofail et al. [16] that illustrates the economic and non-economic impacts of SLS with traditional manufacturing (subtractive manufacturing). It is shown that in the SLS process, industrial needs show SLS is needed in production for manufacturing demands. In contrast to that, two main issues highlighted are handling issues and the overall safety issue of the SLS process. Therefore, the studies related to indoor air exposure is crucial, especially in SLS of AM fields [17], [18].

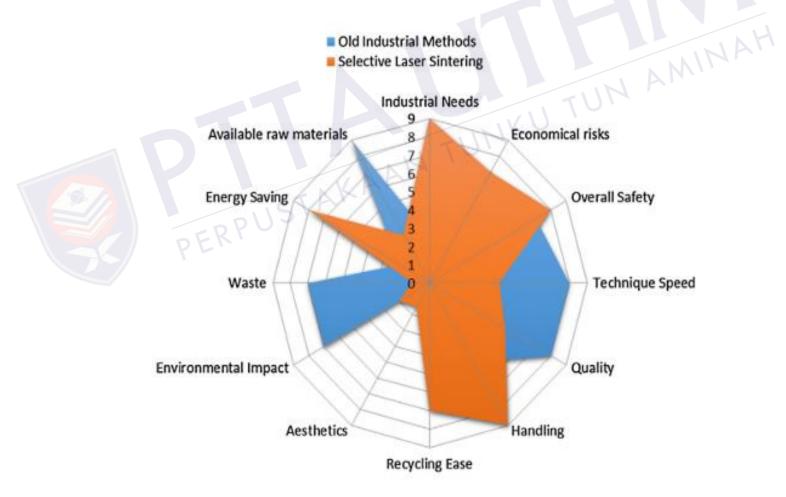


Figure 1.2: Radar chart of old industrial methods and selective laser sintering points adopted from Tofail et al. [13].

Commonly, several factors have been highlighted as the main factors contributing to poor health and productivity: poor IAQ, ergonomics, noise quality, lighting quality, thermal comfort, and ventilation effectiveness. Workers' safety and health will suffer as a result of an unfavourable working environment. Social Security Organization (SOCSO) Malaysia categorised three working environments: outdoor, indoor, and underground [19]. Therefore, pollutant such as respirable and inhalable particulates matter (PM), the volatile organic compound (VOC), and gaseous pollutant identify as the contribution from the SLS printing process in indoor spaces. Figure 1.3 shows deposition potential for PM from varying sizes which can affect human health. PM approximately  $5 - 10 \,\mu$ m will be deposited in the tracheobronchial tree, while PM size  $1-5 \,\mu$ m will be deposited in the respiratory bronchioles. Critically, these kinds of PM can affect and penetrate the lung and enter the bloodstream. PM size smaller than 1  $\mu$ m have similar behaviour to gas molecules and penetrate down to alveoli, and can further into the cell tissue or circulation systems [20]–[22].

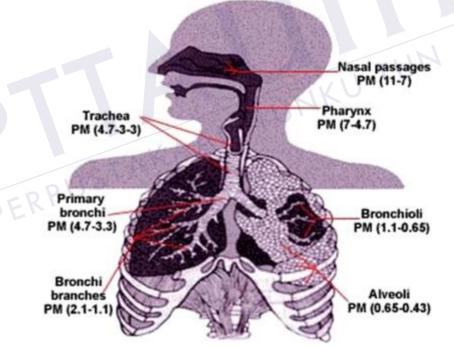


Figure 1.3: Deposition potential of PM and effect on human [23]

Statistical design and analysis of experiments is an effective and commonly used tool in engineering or science investigations. The experiment is performed to understand or improve the system, process or products. In typical methods, one-factorat-a-time (OFAT) employed to monitor the influence of factor and response [24]. Therefore, an increasing number of experiments are needed, leading to high consumption of materials, cost, and time. Multivariate methods such as the Design of Experiment (DoE) have been introduced in recent years to decrease time, effort, and materials. This instrument also allows for collecting a massive quantity of data to reduce the number of tests. DoE also allows the evaluation of several factors at many levels for any experiments. The steps of DoE can be divided into two steps which screening and optimisation design. Screening designs often make use of Plackett-Burman, full or fractional factorial. Subsequently, using RSM, the ideal levels of the most significant parameters picked from the screening design are optimised [25].

### **1.3 Problem Statement**



Selective laser sintering (SLS) technology uses metal or polymer powder to rapidly print complex and high-volume products. In addition, SLS also offers waste material saving up to 90% by using recycled powder or refresh rate [1], [26]. The usage of powder as feedstock materials in SLS processes that generally small size particles (25 to 150 µm) had significantly impacted the environments and operators' health. Particles of this size may be inhaled by the respiratory system and enter the body via the eyes and skin. Later, the worker had a chronic hypersensitivity pneumonitis which reflects directly to SLS working condition [27]. As the SLS process's feedstock, evidence shows that polymers could emit significant amounts of particles and volatile organic compound [28], [29]. However, there is a significant knowledge deficit concerning the risk from inhalation exposure to hazardous substances released into the air during the SLS process, especially in virgin or recycled material [21]. Small particles from SLS powders will stay in the air, but bigger particles will gravitate toward the ground. Additionally, fine particles from the SLS process diminished exceptionally slow, taking at least 2.5 hours to degrade in confined spaces [30]. The process is high-risk, and it includes pre-and post-processing activities such as loading raw material into the machine, pouring, transferring, component removal, and machine cleaning [10].

Even though the SLS sintering process is performed in a closed chamber, operators are still exposed to aerosol and dust during pre- and post-processing phases [31]. According to Chen et al. [30], PM sourced from virgin, recycled and refresh rate powders may be released into the workplace and environments during the manual

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## **APPENDIX C**

## LIST OF PUBLICATIONS/AWARDS

[1] **Damanhuri A.A.M**, Hariri A., Alkahari M.R., Fauadi M.H.F, Bakri S.F.Z, Indoor air concentration from selective laser sintering 3D printer using virgin polyamide nylon (PA12) powder: A pilot study, International Journal of Integrated Engineering, Vol 11, No 5, (2019), pp 140-149 (Scopus)

[2] **Damanhuri A.A.M**, Hariri A, Fauadi M.H.F, Alkahari M.R, Omar M.R, Emission of selected environmental exposure from selective laser sintering (SLS) polyamide nylon (PA12) 3D printing process, Journal of Safety, Health and Ergonomics, Vol 1, No 1, (2019), pp 1-6

[3] **Damanhuri A.A.M**, Subki A.S.A, Hariri A, Tee B.T, Fauadi M.H.F, Hussin M.S.F, Mustafa M.S.S., Comparative study of selected indoor concentration from selective laser sintering process using virgin and recycled polyamide nylon (PA12), IOP Conference Series: Earth and Environmental Science, 373, (2019) (Scopus)

[4] **Damanhuri A.A.M**, Subki A.S.A, Hariri A, Fauadi M.H.F.M, Omar M.R., Lubis A.M.H.S., Total volatile organic compound (TVOC) exposure from recycled polyamide nylon powder during selective laser sintering process, Proceedings of Mechanical Engineering Research Day 2019, pp. 10-11, August 2019 (WoS)

[5] **Damanuri A.A.M**, Hariri A, Ghani S.A, Fauadi M.H.F.M, Hussin M.S.F., Two Level Factorial Design of the Pre-Processing Activities of Polyamide Nylon 12 Powder in Selective Laser Sintering Three Dimensional Printing Process, Journal of Advance research in Dynamical & Control Systems, Vol 12, 08-Special Issue, 2020 (Scopus)

[6] **Damanhuri A.A.M**, Hariri A, Ghani S.A., Mustafa M.S.S, Herawan S.G., Paiman N.A., The Effects of Virgin and Recycled PA12 Powders in SLS Processes on Occupational Exposures, International Journal of Environement Science Development (**Scopus**) Production Process

[7] **Damanhri, A.A.M**, Hariri A., Ghani S.A., Latif K., Optimization of the Refresh Rate and Pre-Processing Paramaters for Selective Laser Sintering of PA12 Powder using Response Surface Methodology, Archieves of Environmental & Occupational Health, Taylor and Francis (WoS) IF: 1.18 (Submitted)

[8] **Damanhuri, A.A.M**, Hariri A, Nordin M, Behavior and Impact of Polyamide Nylon to the Indoor Air Quality, Recent Development in Industrial and Indoor Environment Research Studies, Book Chapter, ISBN 978-967-2916-43-7, Series 1, 2020



Conferences:

[1] 2019 Theory & Technique International Aerosol Conference and Malaysia Air Quality Annual Symposium, 7-10 August 2019, Hotel Balik Pulau, Melaka (Best Presenter Award)

[2] 6th Mechanical Engineering Research Day, August 2019, Faculty of Mechanical Engineering, Universiti Teknikal Malaysia Melaka (UTeM)

Awards:

[1] eResearch & Innovation eCRI 2020, Universiti Malaysia Kelantan (UMK), Optimization Tools of Refresh Rate to Reduce Dust Exposure of PA12 Powder Handling Process, **SILVER Medal** 

[2] International Research and Innovation Symposium and Exposition RISE 2020, Universiti Tun Hussein Onn Malaysia (UTHM), An Optimization Tools of Refresh Rate Towards Dust Exposure of PA12 SLS Handling Process (OP-Refresh), Gold Medal, First Runner Up

[1] **Copyright Registered,** CRLY00026901, Optimization Tools for Refresh Rate and Handling PA12 Powders for SLS Processing (OP-Refresh)

VITA

The author was born in April 20, 1988 in Melaka. He received his primary education at SK Kem Terendak 1 and SK Ayer Hitam, Johor from 1995 to 2000. He continued his secondary school at SMK Kem Terendak, Melaka from 2001 until 2005. Then, he continues his studies in Kolej Mara Kulim Kedah for foundation studies. In 2007, he further his tertiary education in Bachelor of Engineering Technology in Air Conditioning and Industrial Refrigeration from Universiti Kuala Lumpur, Malaysia France Institute (UniKL-MFI). Upon graduation, he worked as Project Engineer in one of contractor specialized in HVAC installation and maintenance involving hospital air conditioning and ventilation system projects. In 2012, he continues his master's degree in Mechanical Engineering from Universiti Tun Hussein Onn Malaysia (UTHM) and graduated in 2014. In 2018, he pursued his doctorate course UTHM in mechanical engineering. During his doctorate studies, the author published several indexed articles and participated in innovation competition. He manages to be won Silver and Gold Medal for both competitions. The author also registered one copyright with MYIPO Malaysia for his optimized model from this study. The author worked as lecturer in Universiti Teknikal Malaysia Melaka (UTeM) since 2015, and recently active in giving training and seminar for short course specialized in HVAC subjects in various vocational training institution in Malaysia. PERPUSTAKAA

