SYNTHESIS OF Al-MCM-41/ZSM-5 COMPOSITE FOR OXIDATION OF NORBORNENE TO NORBORNENE OXIDE

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SYNTHESIS OF Al-MCM-41/ZSM-5 COMPOSITE FOR OXIDATION OF NORBORNENE TO NORBORNENE OXIDE

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Specially dedicated to Mak, Ayah, Along and Angah,
No one else come close to each one of you.
For everything. I thank you.

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ABSTRACT

Composite materials are engineered materials made from two or more constituent material with significantly different physical or chemical properties and the different material work together to give the composite unique properties. The incorporation of Al-MCM-41 and ZSM-5 produced micro-mesoporous composite which very significant to be used in the catalytic study. In this study, the microporous Al-MCM-41 and micro-mesopores Al-MCM-41/ZSM-5 composite have successfully by direct hydrothermal method. The resulting catalyst were characterized by X-Ray Diffraction (XRD), Fourier Transform Infrared (FTIR), Field Emission Scanning Electron Microscopy (FESEM), Transmission Electron Microscopy (TEM), Nitrogen adsorption-desorption isotherm analysis techniques, BET single point, Thermogravimetric Analysis (TGA) and ^{27}Al Magic Angle Spinning-Nuclear Magnetic Resonance (^{27}Al MAS NMR). Catalytic performance on Al-MCM-41/ZSM-5 composites has been carried out by oxidation of bulky molecule norbornene. Higher conversion was observed over Al-MCM-41/ZSM-5 composite 10 wt. % while the selectivity towards norbornene oxide are higher by using the Al-MCM-41/ZSM-5 composite 50 wt. % as catalyst. This is due to the increase of Bronsted acid side which lead to the increased of hydrophilic characteristic of the catalyst and resulting higher formation of norbornene oxide.

ABSTRAK

Komposit merupakan suatu bahan yang terhasil dari penyatuan dua atau lebih bahan yang mempunyai ciri-ciri kimia dan fizikal yang berbeza lalu menghasilkan bahan komposit yang memiliki ciri-ciri yang unik. Penyatuan antara Al-MCM-41 dan ZSM-5 menghasilkan satu bahan komposit berliang mikro-makro yang sangat sesuai untuk diaplikasikan sebagai mangkin. Di dalam penyelidikan yang dijalankan, Al-MCM-41 berilang macro serta komposit Al-MCM-41/ZSM-5 berliang mikro-makro, telah dihasilkan dengan menggunakan proses hidrotermal secara terus. Mangkin yang telah dihasiklan kemudiannya di cirikan dengan menggunakan pelbagai teknik termasuk Pembelauan Sinar-X (XRD), Spektroskopi Infra-Merah Fourier Transform (FTIR), Mikroskopi Elektron Pengimbas (FESEM), Mikroskopi Elektron Transmisi (TEM), Analisis Gravimetri Terma (TGA) dan Resonan Magnetik Nuclear (NMR). Tindakbalas bermangkin dengan menggunakan ZSM-5 serta komposit Al-MCM-41/ZSM-5 sebagai mangkin telah dijalankan ke atas tindakbalas pengoksidaan Norbornene yang merupakan molekul bersaiz besar. Hasil daripada tindakbalas ini, di dapati bahawa peratusan kadar penukaran adalah lebih tinggi apabila Al-MCM-41/ZSM-5 10 wt. % digunakan sebagai mangkin. Namun begitu, kadar pemilihan terhadap norbornene oksida adalah lebih tinggi apabila komposit Al-MCM-41/ZSM-5 50 wt% digunakan sebagai mangkin. Ini adalah di sebabkan oleh peningkatan tapak asid Bronsted yang mengakibatkan peningkatan ciri-ciri hidrofilik pada mangkin serta peningkatan pembentukan norbornene oksida.

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% wt.	Weight percentage
µL	Microliter
2 θ	Bragg angle
^{27}Al MAS NMR	Al Magic Angle Spinning Nuclear Magnetic Resonance
Å	Angstrom
$a_{\circ}$	Unit cell parameter
BET	Brunauer, Emmet and Teller
BJH	Barret-Joyner-Halenda
CTABr	Hexadecyltrimethylammoniumbromide
Cu K_{α}	X-ray diffraction from copper K_{α}
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier Transform Infrared
GC-MS	Gas Chromatography- Mass Spectrometry
GS-FID	Gas Chromatography – Flame Ionization Detector
Hz	Hertz
IUPAC	International Union of Pure and Applied Chemistry
MCM-41	Mobil Crystalline Material
mg	Milligram
min	Minute
mL	Milliliter
RHA	Rice husk ash
TEM	Transmission Electron Microscopy (TEM)
TGA	Thermogravimetric Analysis
XRD	X-Ray Diffraction
δ	Chemical shift

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Catalyst can be defined as a substance that accelerates a reaction but undergoes no net chemical changes (Atkins and de Paula, 2002). There are various types of catalysts used in reactions and heterogeneous catalysis is the main type. In the fine chemical industries, most of the oxidation processes which are being carried out in the gas and liquid phases is widely used in bulk chemicals manufacture. The catalytic oxidation of olefins to epoxides (oxiranes) is of great importance due to epoxides products are valuable intermediates for a wide variety of bulk chemicals, polymers, and fine chemicals. Epoxides can be easily converted into dialcohols, aminoalcohols or ethers, therefore the formation of chiral epoxides is a very important step in the synthesis of natural product.

Porous material has been intensively study with regards to technical application as catalyst and catalyst support. According to the IUPAC definition, porous material are divided into three classes; microporous (pore size < 2 nm), mesoporous (pore size of 2 to 50 nm) and macroporous (> 50 nm) material. Among the family of microporous materials, the best known members are zeolites which have a narrow and uniform micropore size distribution due to their crystallographically defined pore system. However, microporous zeolite having pore aperture sizes smaller than 7 Å cannot catalyze the oxidation of bulky organic molecules due to the inaccessibility to the active sites located inside the micropores (Igarashi *et al.*, 2005).

ZSM-5 zeolite, which was first developed in 1972 by Mobil, has a regular channel system, i.e. straight circular channels (5.4 Å-5.6 Å) interconnecting with sinusoidal and elliptical channels (5.1 Å-5.4 Å) (Wang *et al.*, 2007). The properties that make ZSM-5 suitable for industrial applications are its extremely high thermal and acidity stability, high selectivity, and high activity in certain catalytic conversion especially in the case of shape-selective catalyst (Wang *et al.*, 2007). Unfortunately, zeolites present severe limitations when large reactant molecules are involved, especially in liquid-phase systems as is frequently the case in the synthesis of fine chemicals, due to the fact that mass transfer limitations are very severe for microporous solids. Attempts to improve the diffusion of reactants to the catalytic sites have so far focused on increasing the zeolite pore sizes, on decreasing zeolite crystal size, or on providing an additional mesopore system within the microporous crystals.

Researches on mesoporous silicas and related materials are important fields of recent material science. Especially MCM-41and its analogues are actively studied because of their high potentials for various applications. The inventions of mesoporous material by Kresge *et al.* (1992), Kuroda (1996), and Inagaki *et al.* (2004) belonging to Mobil, Waseda and Toyota research groups, respectively have lead to the intensive studies for developing highly active catalytic materials. MCM-41, which is a member of mesoporous molecular sieves family have been recently discovered by researcher at Mobil Corporation (Chen *et al.*, 1999). MCM-41 stands for Mobile Composition of Matter No. 41 possessed highly ordered hexagonal array of unidimensional pore with very narrow pore size distribution. The narrow pore size distribution adjustable in the 15-100 Å range and an extremely high surface area makes these materials promising candidates as catalysts or catalyst supports, absorbent and as a host.

The main disadvantage of MCM-41 is that its wall is amorphous, thereby leading to its lower mechanical, thermal and hydrothermal stability (Chen *et al.*, 2009). Apart from that, MCM-41 also does not exhibit comparable catalytic properties to those of microporous silicate such as ZSM-5 and zeolite Y which have abundant, uniform micropores structures and strong acidities which lead them to possess crystalline structures and higher stability. Unfortunately, the microporous silicates are not appropriate for effectively reacting with large molecules because of the limitation of their small pore size.

Recently, the composite materials have been a significant concern as to developing materials with unique properties. Composite material or composite is defined as engineered material made from two or more constitual materials with significantly different physical and chemical properties which remains separate and distinct on a macroscopic level within the finished structure. There are many studies had been done in developing composite materials which includes the usage of mesoporous and microporous materials. Basically, the meso-microporous materials provide bimodal pore systems and combine the benefits of each pore size regime which could potentially improve the efficiency of zeolite catalysis.

Thus, great interest has been generated in the synthesis of bimodal composite materials to combine the performances of both mesoporous and microporous molecular sieves. Jacobsen *et al*., (2000), have reported the usage of mesoporous carbon blacks as templates to prepare nanosized zeolites, which possess micro-mesoporous properties by using one-step crystallization. Kloetstra *et al*., (1996), on the other hand has reported the preparation of zeolite faujasite coated with a thin layer of mesoporous MCM-41 prepared by a one step synthesis. Huang *et al*., (2000), reported the preparation of ZSM-5/MCM-41composites by using a dual template method through a process of a two-step crystallization. Guo *et al.*, (2001), synthesized a series of Beta/MCM-4l composites with various crystallinities through a two-step crystallization process.

Basically, an alkene can be oxidized to an epoxide by a peroxyacid (Bruice, 2004). The epoxidation of an alkene is a concerted reaction, where all the bond-forming and bond-breaking process takes place in a single step. The setback of homogeneous epoxidation of alkene reaction is the usage of expensive peracids as oxidants apart of the formation of large amount of effluent (Blasco *et al.*, 1995). In order to overcome this problem, this study is focused on the catalytic performance of

Al-MCM-41/ZSM-5 composite as a heterogeneous catalyst in the epoxidation reaction by using hydrogen peroxide as an oxidant.

MCM-41 is a member of a family of mesoporous molecular sieves that had been discovered by researcher at Mobil Corporation (Chen *et al.,* 1999). Purely siliceous MCM-41 is amorphous and has no Bronsted acidity. The isomorphous substitution of Si by trivalent cation such as Al by the isomorpous substitution of the aluminium for silicon atoms during hydrothermal synthesis or secondary synthesis creates moderately acidic sites (Zanjanchi and Asgari, 2004). The synthesis of Al-MCM-41 by a direct method exhibits low hydrothermal stability especially at relatively low Al-content. (Adjdir *et al.,*2009a). However, Al-containing MCM-41 has relatively low acidity as compared to that of the zeolite ZSM-5. Therefore, the improvement on the acidity of this mesoporous material is required.

Zeolite, especially the ZSM-5 zeolite is very much of interest due to its high acid strength, pore shape, selectivity and also stability. The number and the strength of acid sites dictated by distribution of aluminum ions in ZSM-5 crystal lattice are responsible for its catalytic activity towards various reactions (Sahoo *et al.*, 2001). The main inconvenient of ZSM-5 for many applications is its micropore size which limit the accessibility to substrates of small kinetic diameter which is smaller than 7 Ǻ (Trong *et al*., 2003).

Green technology or the use of environmental friendly chemical process is now gaining popularity because of the demand from consumer as well as strict environmental regulation. Thererfore, application of the heterogeneous catalyst is very wide nowadays as compared to the application of the homogeneous catalyst. In view of the pore size limitations of microporous zeolites and low acidity mesoporous silica materials, it is of interest to prepare a composite catalyst that would combine the Bronsted acidity of crystalline zeolite with the regular pore size of amorphous mesoporous material. In present study, mesoporous Al-MCM-41 material is composite with ZSM-5 in order to enhance the acidity, thermal stability as well as catalytic property of MCM-41 in the oxidation of norbornene. For industrial application, zeolites are known as promising component in heterogeneous acid catalyst. The mass transfer to the active site of zeolite inside the micropores is quite important to industrial aspect. However, the efficiency of zeolite is limited because of its microporosity, leading to the deficiency in diffusion limitation throughout the crystal, resulting in saturation of catalyst efficiency and performance (Ogura *et al.*, 2007). Apart from that, Al-MCM-41 also have setback which is due to its amorphous character which result in comparison with microporous zeolite in lower acidity and hydrothermal stability. Therefore, the application of this type of mesoporous aluminosilicate as acid catalyst for processing of bulky compounds is limited (GonÇalves *et al*., 2008).

To overcome this limitations, the study is being carried out to combine mesoporosity of the mesoporous aluminosilicate with the acidity and stability of microporous zeolite. In the present study, the Al-MCM-41 and Al-MCM-41/ZSM-5 composite is being synthesized. This is because the incorporation between both Al-MCM-41 and ZSM-5 may be a good attempt as to improve the diffusion of reaction to the catalytic site of zeolite by providing additional mesopores system within the microporous crystal, allowing larger molecules to enter the pore system, to be prossed there and to leave the pore system again (Taguchi and Schuth, 2005). This improvement of chemical and physical properties of the material makes Al-MCM-41/ZSM-5 composite a better catalyst as compared to Al-MCM-41 or ZSM-5 alone. The comparison between both microporous/mesoporous composite with incorporation of different weight percentage of ZSM-5 catalyst is very important to distinguish which is a better catalyst toward oxidation of bulky norbornene, known as bicyclo [2.2.1] hept-2-ene to norbornene oxide.

1.2 Objective of Study

The objectives of this study are:

i. To synthesize mesoporous Al-MCM-41 directly by hydrothermal method with initial molar composition of 6 SiO_2 : CTABr : 1.5 NaO : 0.15 $(NH_4)_2O$: 250 H_2O with Si/Al = 30.

ii. To synthesize Al-MCM-41/ZSM-5 composite with different ratio of ZSM-5 weight percentage by using direct synthesis through two step re-crystallization process.
iii. To characterize the physicochemical properties of the synthesized Al-MCM-41 and Al-MCM-41/ZSM-5 composites.
iv. To determine the catalytic performance of Al-MCM-41/ZSM-5 composite with different wt. % of ZSM-5 in the oxidation of bulky molecule norbornene to norbornene oxide with hydrogen peroxide as oxidant.

1.3 Scope of Study

The conceptual framework of the study is shown in Figure 1.1. This research is carried out to synthesize mesoporous Al-MCM-41 with the sol-gel method with initial molar composition of 6 $SiO_{2:}$ CTABr: 1.5 NaO: 0.15 $(NH_4)_2O$: 250 H_2O with Si/Al = 30. The silica source used in synthesizing the Al-MCM-41 is rice husk ash with SiO_2 content of 93%. The synthesis of Al-MCM-41/ZSM-5 composite is done by using the same method as in the preparation of the Al-MCM-41 but with addition of microporous ZSM-5 zeolite. In the study, the commercial ZSM-5 zeolites were added in two different ratios of weight percentage which are 10 wt.% and 50 wt.% respectively.

The BET single point method is used to determine the surface area of Al-MCM-41, Al-MCM-41/ZSM-5 composite. 10 wt.% and also Al-MCM-41/ZSM-5 composite 50 wt.%. The Nitrogen Adsorption and desorption Isotherm is used to determine the specific surface area as well as to monitor and determine the porosity or the pore texture of the Al-MCM-41/ZSM-5 composite.

The structure characterization of the sample is characterized by using Fourier Transform Infrared Spectroscopy (FTIR) and X-Ray Diffraction Spectroscopy (XRD). The Field Emission Scanning Electron Microscopy (FESEM) is being used to determine the surface morphology of the produced Al-MCM-41 and Al-MCM-

41/ZSM-5 composite as well as the commercial ZSM-5. Transmission Electron Microscopy (TEM) on the other hand is being employed to analyze the pore shape and pore size of Al-MCM-41/ZSM-5 composite. The thermal stability of Al-MCM-41 and Al-MCM-41/ZSM-5 composite is determined by using Thermogravimetric Analysis (TGA). Lastly, the ^{27}Al MAS NMR is used to characterize the nature of aluminum species (tetrahedral or octahedral) in the composite.

Both catalysts (Al-MCM-41/ZSM-5 composites with different wt.% of ZSM-5) performances are tested in the oxidation of bulky norbornene with hydrogen peroxide (H_2O_2) as oxidant. The reaction was carried out in a batch reactor and the products obtained were analyzed by gas chromatography (GC). The products obtained from oxidation reaction were analyzed qualitatively by GC-MS and quantitatively by GC-FID equipped with a HP-5 column.

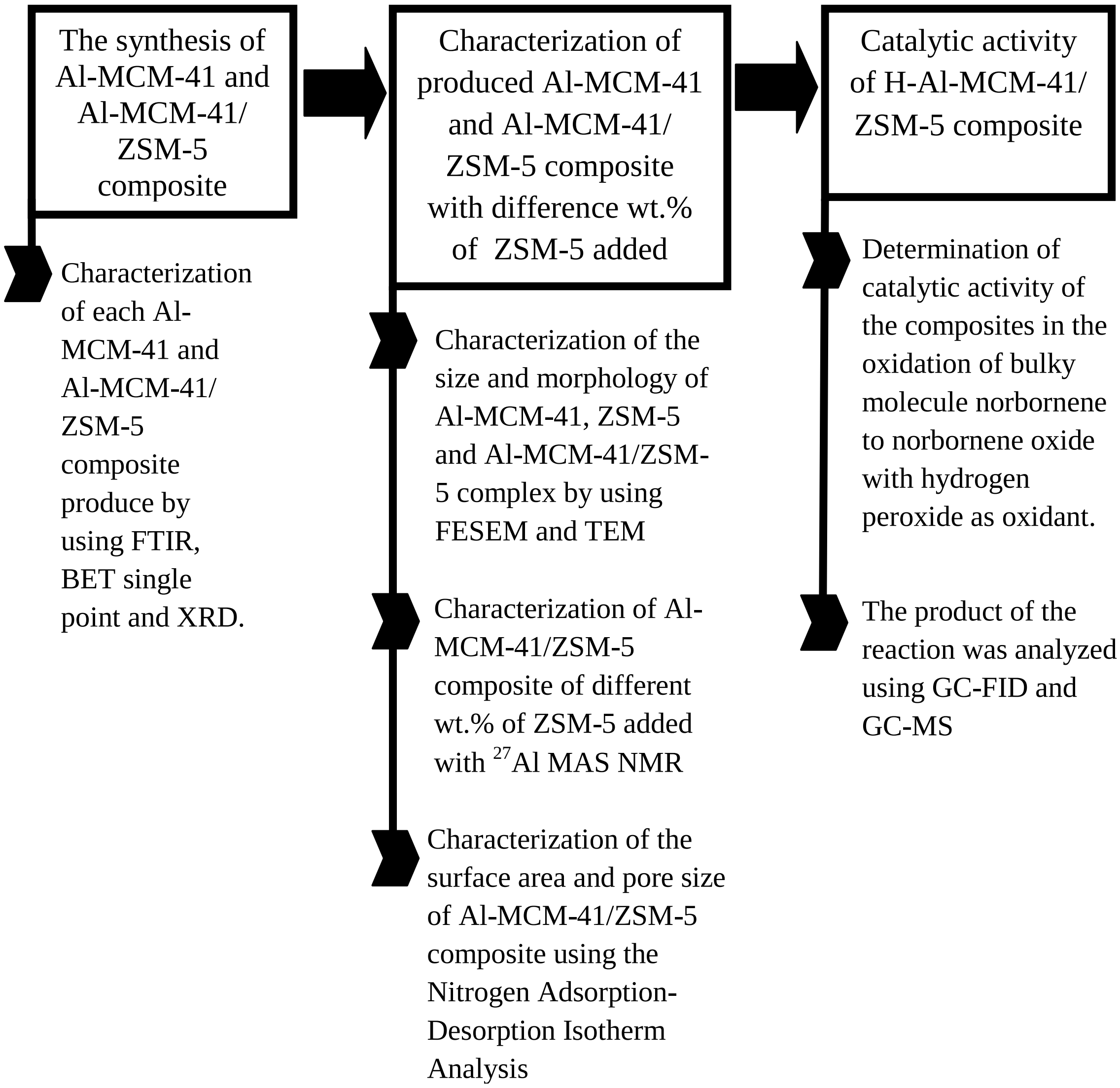

Figure 1.1: Conceptual Framework

EFFECT OF CUTTING PARAMETERS ON THE HOLE DIAMETER AND SURFACE ROUGHNESS FOR DRY DRILLING OF ALUMINUM ALLOY 6061

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To my beloved family Father, Mother, Aina Fairiza, Amni Fasihah, Amsyar Fahim, Brothers and Sisters. Thank for all your support .

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ABSTRACT

Green manufacturing and sustainability are becoming very important issues in today technologies. Besides, dry machining represents cost saving opportunities related to cooling lubrication. In addition, drilling holes is one of the major manufacturing processes which include a wide range of products. The objective of this work was to find out the optimum cutting parameters to optimize the surface roughness of the hole and its diameter accuracy in the dry drilling process based on the selected setting parameters. The selected material for this study was Aluminium alloy 6061. The selected cutting parameters for this study were the cutting speed, feed rate, and the hole depth. The effect of these parameters on the hole diameter accuracy and its surface roughness are measured and analyzed. Experiments were conducted based on Design of Experiment (DOE), and the analysis of variance (ANOVA) is employed to find the optimal level and to analyze the effect of the drilling parameters on surface roughness and hole diameter accuracy values. Result shown that cutting parameters have the significant effect to the hole diameter and for surface roughness all the three parameters have the significant effect. Confirmation tests were conducted in order to validate the test result.

ABSTRAK

Persekitaran yang baik dan berkekalan adalah amat penting dan harus berterusan mengikut arus teknologi pada masa kini. Pemesinan kering dapat mengurangkan kos yang berkaitan jika dibandingkan apabila menggunakan pelincir semasa proses pemesinan. Penggerudian lubang adalah salah satu proses utama dalam penghasilan produk di mana ianya meliputi hampir keseluruhan dari pembuatan sesuatu produk itu. Tujuan utama penghasilan kertas kerja ini ialah untuk mendapatkan kesan pemesinan pada tahap optimum dengan menggunakan parameter yang berkaitan untuk permukaan akhir dan diameter lubang yang terbaik dalam pemesinan. Bahan kerja yang dipilih ialah aluminium aloi 6061. Parameter yang dipilih dalam kertas kerja ini ialah kelajuan pemotongan, kadar suapan dan juga kedalaman pemotongan. Kesan hasil dari parameter yang digunakan terhadap permukaan akhir dan lubang diameter diukur dan dianalisa. Eksperimen dilakukan secara statistik dengan menggunakan analisa variasi (ANOVA) bagi mendapatkan nilai yang terbaik untuk parameter dalam proses penggerudian terhadap permukaan akhir dan diameter lubang. Keputusan menunjukkan kelajuan pemotongan mempengaruhi diameter lubang dan bagi permukaan akhir, ketiga-tiga parameter yang digunakan mempengaruhinya. Pengesahan ujikaji dilakukan bagi mengesahkan keputusan ujikaji.

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LIST OF ABBREVIATIONS AND SYMBOLS

ANN	-	Artifical neural network
ANOVA	-	Analysis of variance
ANSI	-	American National Standard Industries
BS	-	British Standard
CAD	-	Computer aided design
CAM	-	Computer aided manufacturing
CCD	-	Central composite design
CNC	-	Computer numerical control
CFRP	-	Carbon fiber reinforced plastic
CHM	-	Chemical machining
CMM	-	Coordinate measuring machine
DIN	-	Deutsch International Standard
DOE	-	Design of experiment
ISO	-	International Standard of Organization
IT	-	International Tolerance Grade
JIS	-	Japanese International Standard
RPM	-	Revolution per minutes
HSS	-	High speed steel
MANOVA	-	Multi analysis of variance
SEM	-	Scanning Electron Microscopy
S/N	-	Signal to noise ratio
TiN	-	Titanium nitride
R_a	-	Arithmetical mean value of surface roughness
Rmax	-	Maximum height of the irregularities of surface roughness
D/d	-	Diameter

T	-	Tolerance
Y	-	Ordinates of the profile curve
3D	-	3 Dimensional
y	-	Response
ε	-	Error
η	-	Expected response
^{o}	-	Degree/Angle

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CHAPTER 1

INTRODUCTION

1.1 Overview

Drilling processes are widely used in the aerospace, aircraft and automotive industries (Mustafa Kurt *et.al,* 2009). Although that non-traditional machining methods have improved in the manufacturing industries in response to new and unusual machining requirement that could not be satisfied by conventional methods. Non-traditional machining including ultrasonic machining, abrasive water jet cutting, electrochemical machining (ECM), and chemical machining (CHM) are some of the example but conventional drilling still remains one of the most common machining processes.

In drilling processes, cutting fluids are used to lubricate the process and reducing the temperature that contributes of wear and tear to the cutting tool. Aluminium alloys widely used for automotive and aerospace industries which durability, strength, and light weight are desired and these materials subjected to machining operations where the criterion of minimization of lubricant or coolant use is becoming more topicality. Manufacturer have desired to work without any lubricant because of reasons such as the cost of using it, supply and maintenance of the lubricant, hazard arising from the lubricant and the disposal of used lubricant, therefore an alternative methods of machining is a dry machining.

A statistical technique, fractional factorial experiments and analysis of variance (ANOVA), has been employed to investigate the influence of cutting parameters on the hole diameter accuracy and surface roughness quality. Factors affecting surface roughness were determined and thus conclusions were drawn about optimizing all the cutting parameters for better hole diameter accuracy and surface roughness quality. Many factors affect hole diameter and surface roughness quality, which can be divide into controllable and non-controllable (Shashidhar Madiwal, 2006).

Controllable Factors:

Speed Feed Rate
Hole Depth
Drill Geometry
Material of Cutting Tool

Non-Controllable Factors:

Machine Accuracy
Tool Geometry
Material Properties

Taking the above factors into consideration, experiment was conducted to determine the best combination factors to obtain optimum hole diameter and surface roughness quality. A methodology was adopted to conduct these experiments in organized method which was the Design of Experiment (DOE) and the data from the experiment was analyzed.

1.2 Objective

a. The main objective of this project is to study the effect of speed and feed rate, also hole depth in dry drilling process of aluminum 6061 alloy for hole diameter accuracy and surface roughness quality.
b. To establish empirical formula for the cutting parameters for aluminum alloy 6061 by utilizing Design of Experiment (DOE) approach.

1.3 Scope of Project

The scope of work is clearly define the specific field of the research and ensure that the entire content of this thesis is confined the scope. It is begun with the literature review. The next step is to perform experimental studies on dry drilling process of aluminum alloys to determined the significant parameters affecting two responses for surface roughness quality and hole diameter accuracy. The result of the experiment for the hole diameter and surface roughness quality are analyzed under varying cutting speed, feed rates, and hole depth with low and high values setting.

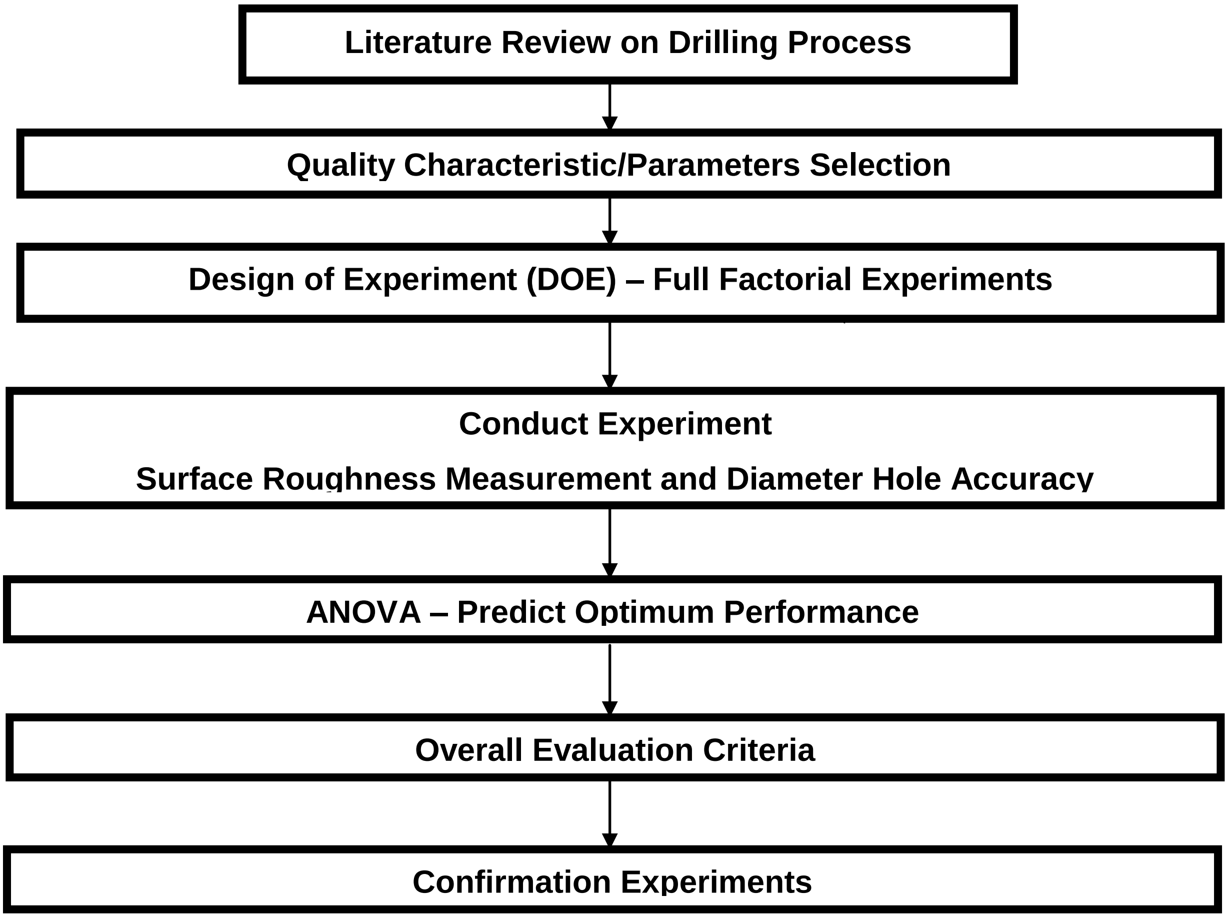

Figure 1.1: Flow chart represents the scope of work

1.4 Problem Statement

Based on the literature review on the past research on drilling operations, the following items have been revealed on the processes;

a. Cutting fluids are used to lubricate the process and reduce the temperature for the surface roughness quality.
b. Analysis of drilling quality is focusing on composite material, burr height and burr size and also tool wear.
c. Most of the researcher used Taguchi method and with other methods to determine the optimum parameters.