PREPARATION AND CHARACTERIZATION OF CERAMIC PORCELAIN PREPARED THROUGH SLIP CASTING

NURUL'AIN BINTI HARIS

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

C Universiti Teknikal Malaysia Melaka



UNIVERSITI TEKNIKAL MALAYSIA MELAKA

PREPARATION AND CHARACTERIZATION OF CERAMIC PORCELAIN PREPARED THROUGH SLIP CASTING

This report submitted in accordance with requirement of the University Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Materials Engineering) with Honours.

by

NURUL'AIN BINTI HARIS

FACULTY OF MANUFACTURING ENGINEERING

C Universiti Teknikal Malaysia Melaka

DECLARATION

I hereby, declared this report entitled "Preparation and Characterization of Ceramic Porcelain Prepared through Slip Casting" is the results of my own research except as cited in references.

Signature	:
Author's Name	: Nurul'Ain Binti Haris
Date	: 10 th April 2009



APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Materials Engineering) with Honours. The member of the supervisory committee is as follow:

(Signature of Supervisor)

.....

(Official Stamp of Supervisor)



APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Materials Engineering) with Honours. The member of the supervisory committee is as follow:

(Signature of Principal Supervisor)

.....

(Official Stamp of Principal Supervisor)

(Signature of Co-Supervisor)

.....

(Official Stamp of Co-Supervisor)

C Universiti Teknikal Malaysia Melaka

ABSTRACT

Porcelains made of ball clay, feldspar, kaolin and quartz were investigated to study the effect of adjustment of quartz content on the bending strength. These raw materials are mixed by ball milling and formed by slip casting. The bending strength for Batch A is the highest which is 22.51 MPa followed by batch C which is 12.89 MPa and batch B, 8.21MPa. Batch A with the highest quartz content results in lowest reading of total linear shrinkage after drying and sintering which is 24%. A decreasing content of quartz leads to increasing shrinkage. The shrinkage of porcelain increases from bisque firing temperature of 900°C to final sintering temperature of 1250°C. The analysis methods are done to determine the morphologies of the porcelain ceramics using Scanning electron microscopy (SEM) and X-Ray Diffraction (XRD). In XRD analysis, the existence of quartz as SiO_2 is more compared to the existence of mullite in Batch A meanwhile in SEM analysis, the amount of pores in Batch A is less than Batch B and batch C. These analyses have provided the information that increasing in quartz content leads to decreasing in pores amount in a porcelain body and this finding is then correlated to the strength and shrinkage of the body.

ABSTRAK

Porselin diperbuat daripada ball clay, feldspar, kaolin dan quartz telah diselidiki untuk mengetahui kesan pengubahan kandungan quartz ke atas kekuatan kelenturannya. Bahan-bahan mentah ini digaul menggunakan penggilingan bebola dan dibentuk dengan teknik penuangan. Kekuatan kelenturan bagi batch A ialah yang tertinggi iaitu 22.51 MPa diikuti oleh batch C dengan 12.89 MPa dan batch B, 8.21MPa. Batch A mempunyai kandungan quartz yang tertinggi menghasilkan bacaan total linear pengecutan selepas pengeringan dan pensinteran yang terendah iaitu 24%. Pengurangan kandungan quartz mengakibatkan peningkatan pengecutan. Pengecutan porselin meningkat dari pembakaran bisque pada 900°C ke pensinteran terakhir pada 1250°C. Analisis yang dilakukan bagi menentukan morfologi porselin menggunakan Scanning electron microscopy (SEM) dan X-Ray Diffraction (XRD). Dalam analisis XRD, kewujudan quartz sebagai SiO2 lebih berbanding kewujudan mullite dalam Batch A sementara dalam analisis SEM, bilangan pori dalam Batch A kurang daripada Batch B dan Batch C. Analisis -analisis ini telah menyediakan informasi bahawa kandungan quartz memimpin kepada penurunan bilangan liang dalam porselin dan penemuan ini dikaitkan dengan hubungan antara kekuatan dan pengecutan porselin.

DEDICATION

To my beloved mother and family.

ACKNOWLEDGEMENT

First and foremost, I would like to express my fullest gratitude to Dr Azizah binti Shaaban of University Technical Malaysia Malacca. She, as my Project Sarjana Muda supervisor had provided me not only her valuable knowledge, but also her time to help us to overcome any difficulty during the study. I would like to thank Dr Jariah binti Mohamad Juoi, my lecturer on Ceramics Material subject and Dr. Warikh, my lecturer on guiding me and helping me to understand more on my research. Thanks to my family and friends for helping me during the time of difficulty.

TABLE OF CONTENTS

ABSTRACT i		
ABSTRAK		
DEDICATION		
ACKNOWLEDGEMENT		iv
TAB	LE OF CONTENT	V
LIST	OF TABLES	viii
LIST	OF FIGURES	ix
LIST	OF ABBREVATIONS, SYMBOLS & NOMENCLATURE	xii
1. IP	NTRODUCTION	1
1.1	Introduction to Ceramics	1
1.2	Background	3
1.3	Problem Statement	6
1.4	Objective	6
1.5	Scope	6
2. L	ITERATURE REVIEW	8
2.1.	Porcelain	8
2.1.1	A Ceramic Porcelain Phase Diagram (Al ₂ O ₃ -SiO ₂)	8
2.1.2	Properties of Porcelain	10
2.1.3	Categories of Porcelain	11
2.2	Strength Considerations	12
2.2.1	Hypotheses from Previous Research	12
2.3	Constituents of Porcelain	14
2.3.2	Ball clay	14
2.3.3	Silica	15
2.3.4	Feldspar	16
2.3.5	Kaolin	16
2.4	Body Formulation of Porcelain	17
2.5	Application of Porcelain	19

2.5.1	Insulators	19
2.5.2	Tableware	20
3. M	ETHODOLOGY	21
3.1 I	ntroduction	21
3.2 N	Material Selection and Compositions	22
3.3	Formation of Ceramics Porcelain	24
3.3.1	Ball Milling	25
3.3.2	Slip Casting	28
3.3.3	Drain Casting	30
3.3.4	Drying Process	31
3.3.5	Sintering Process	33
3.4	Sample Dimensioning	36
3.5	Properties Evaluation	36
3.5.1	Drying And Firing Shrinkage Test	37
3.5.2	Flexure Test	38
3.5.3	Scanning Electron Microscope	40
3.5.4	X-ray Diffraction	41
4. R	ESULT AND DISCUSSION	42
4.1	Particle Size Evaluation	42
4.2	Slip Properties	44
4.3	Solid Casting	46
4.3.1	Drying Of Porcelain Ceramics	47
4.3.2	Sintering Of Porcelain Ceramics	49
4.3.3	Shrinkage	51
4.3.4	Mechanical Properties	54
4.3.5	X-Ray Diffraction (XRD) Analysis	57
4.3.6	Scanning Electron Microscopy (SEM) Analysis	60
4.4	Drain Casting	61
4.5	Defects on Products	63
4.5.1	Holes and Voids	63
4.5.2	Cracks	64
4.5.3	Warping	65

Vi C Universiti Teknikal Malaysia Melaka

5. CONCLUSION AND RECOMMENDATION

5.1	Conclusion	66
5.2	Recommendation	67

REFERENCES

APPENDICES

A Gantt chart PSM

LIST OF TABLES

1.1	The general classifications of traditional ceramics	3
1.2	Timeline of selected ceramic and glass developments	5
	(ACerS, 1920)	
2.1	Chemical characteristics of the raw materials used in the porcelain	18
	body in wt%.(Vieira and Monteiro,2007)	
3.1	Compositions of raw materials in Batch A,B and C	22
3.2	The parameters of ceramic porcelain preparation	23
3.3	Apparatus in Ball Milling	22
4.1	The parameters considered to determine SG.	45
4.2	Dimensions of product for drying process	47
4.3	Overall dimensions of product after drying process	48
4.4	Dimensions of product after sintering process	50
4.5	Overall dimensions of product after sintering process	50
4.6	Linear drying shrinkage of specimens as a function of time	52
4.7	Total linear shrinkage, S_t after drying and firing of specimens as a	54
	function of temperature	
4.8	MOR of silica content of (a) 19.975%, (b) 16.875% and (c)	55
	18.75% (d) overall MOR	
4.9	Dimensions of crucible product within drying period	62
4.10	Overall dimensions of crucible product after sintering process	62

LIST OF FIGURES

1.1 Categories of ceramics

2.1	The Al ₂ O ₃ -SiO ₂ phase diagram (Henkel and Pense, 2002)	9
2.2	Diagram of the system MgO-Al2O3-SiO2. (Greenhut, 2001)	10
2.3	Porcelain Insulators (Anonymous, 2008)	20
2.4	Porcelain Table-Ware (Anonymous, 2008)	20
3.1	The process flow of sample preparation	21
3.2	Front view of assembled mold	23
3.3	The lower half	23
3.4	The upper half	24
3.5	The mold measurement	24
3.6	Dimensions of slip casting product. The wedge acts as slip	24
	reservoir	
3.7	Schematic Diagram of Rotating Pot, the Mixture and Balls during	25
	Ball Milling Operation	
3.8	The Ball Mill Machine	26
3.9	The mixture is stirred with the ball mills	27
3.10	The slip is weighed to get the required specific gravity	28
3.11	The Steps in Solid Slip Casting Using A Plaster Of Paris Mold	28
	(Callister, 2005)	
3.12	The slip is injected into the mold	29
3.13	The spills on the mold are cleaned using scrapper	29
3.14	The final product of solid casting	30
3.15	The slip is drained out of the crucible mold after given period	31
3.16	Several stages in the removal of water from between clay	32
	particles during the drying process. (a) wet body, (b) partially	
	dry body, (c) completely dry body (Callister, 2005)	
3.17	Drying is done by placing the cast part on a tray at room	33
	temperature	

3.18	The Sintering Machine	33
3.19	Typical Firing/Sintering Curves with Corresponding Reactions	34
	Obtained For Clay-Bearing Ceramic Composition	
	(Greenhut, 2001)	
3.20	The specimens and crucibles are arranged in a furnace,	35
	ready to be sintered.	
3.21	The specimens and crucibles are arranged in a furnace, waiting	35
	to be sintered	
3.22	The schematic flow diagram of bisque firing process with	35
	increasing heating rate of 10°C/min	
3.23	3-Point Flexure Test	39
3.24	Two Bend Test Method for Brittle Materials.	39
	(a) Three point bending (b) Four point bending	
	(Kalpakjian and Schmid, 2006).	
3.25	The Scanning Electron Microscope (SEM)	38
41	Particle size of (a) hall clay (b) feldspar (c) kaolin and (d) silica	44
4 2	Specific Gravity of batch A B and C Batch A has the highest	45
	viscosity	10
4.3	The relation between amount of water and specific gravity of	45
	slip. As water increases, SG decreases.	
4.4	Schematic diagram of the slip cating system (a) initial system,	46
	(b) after the formation of a thin cast (Rahaman,2003)	
4.5	The relation between dimension and drying period. Dimension	46
	decreases as drying period increases	
4.6	Change in moisture content in ceramics during drying	46
	(King,2002)	
4.7	The dimensions of products decrease as temperature increases	51
	due to shrinkage	
4.8	Illustration shows how removal of material from the area	51
	between particles into the pore lead to shrinkage and	
	densification((Barsoum,2002).	
4.9	Linear drying shrinkage measurement as a function of time.	53
	Optimum shrinkage of drying is obtained on third day and	

C Universiti Teknikal Malaysia Melaka

maintains on fourth day(Barsoum,2002))

4.10	Shrinkage of the specimens as a function of batch	
4.11	The correlation of silica content and flexural strength. As	57
	silica content increases, flexural strength increases	
4.12	The XRD analysis on (a) batch A, (b) batch B and (c) batch C	58
4.13	The micrographs of SEM analysis on batch A, B and C	61
4.13	The slip is drained out of the crucible mold after given a	57
	period of casting time	
4.14	Hole in a porcelain body due to improper and inadequate filling	63
4.15	Pipes is evident when the body is cut in half or after fracture.	64
	(Barsoum, 2003)	
4.16	Cracks in a porcelain body due to thermal shock	64
4.17	Warping of a porcelain body due to incorrectly removal	65
	from mold	

LIST OF ABBREVATIONS, SYMBOLS & NOMENCLATURE

ACerS	_	American Ceramic Soceity
ASTM	_	American Society for Testing & Materials
MOR	_	Modulus of Rupture
SEM	_	Scanning Electron Microscopy
SG	_	Specific Gravity
XRD	_	X-Ray Diffraction

$AL_2O_3.2SiO_2.2H_2O$	_	Ball Clay
K ₂ O.AL ₂ O ₃ .6SIO ₂	_	Feldspar (orthoclase)
AL ₂ Si ₂ O ₅ (OH) ₄	_	Kaolin
SiO ₂	_	Silica or quartz

CHAPTER 1 INTRODUCTION

1.1 Introduction to Ceramics

Ceramics are defined as solids composed of compounds that contain metallic and non metallic elements and the atoms in the compound are held together with strong atomic forces either ionic or covalent bonds (Budinski, 2005). Ceramic engineers and materials engineers are the people who design the processes of fabricating ceramics products. They also create new types of ceramic products and find different uses of ceramic products in everyday life. Ceramics are essential to our daily lifestyle and their applications of products are widely used. Ceramics materials include things like tile, bricks, plates, glass, and toilets. Ceramics can be found in products like watches where it is used as the quartz tuning forks which function as the time keeping devices in watches. Other than that, its application can be found in automobiles where the sparkplugs and engine parts in race cars are made of ceramics. They can also be found on space shuttles and appliances which the enamel coatings are made of ceramics. Ceramics can be dense or lightweight depending on their method of formation. Ceramics usually shows excellent strength and hardness properties. However, they have one significant drawback which is their brittleness but this problem has been solved by the development of new materials such as composites.

Ceramics can be divided into three categories; traditional ceramics or clay based ceramics, advanced ceramics and porous ceramics. Clay based ceramics are produced from minerals mined directly from the earth and falls into three commonly recognized

1

groups; clay, silica, and feldspar. They are used in a variety of applications including porcelain, refractory and structural clay products. Porous ceramics are developed for their porous natures that are hydrophilic (water loving) and provide capillary wicking and transport of polar substances such as water. The advantages of porous ceramics are that they are not only mechanically strong but inert and can be cleaned and reused in many process applications. The advanced ceramic is the materials that are well defined and their controlled properties are produced from nearly chemically and phase pure starting materials. They can be classified into three categories; oxides ceramics, non-oxides ceramics and composite ceramics. The examples of oxide ceramics are alumina and zirconia. Carbides, borides, nitrides and silicates are in non- oxide ceramics and ceramic based composite are the particulate reinforced and in combinations of oxides and non oxides. The categories of ceramics are as shown in **Figure 1.1**.



Figure 1.1: Categories of ceramics

The main emphasize in this research is the traditional or clay based ceramics as ceramic porcelain lies in this category. The general classifications of traditional ceramics are as shown in **Table 1.1**.

Classifications	Descriptions	
Pottery	Used as a generic term for ceramics that contains clay and is not used	
	for structural, technical or refractory purposes.	
Whiteware	Ceramic ware that is white, ivory or light gray in color after firing.	
	Whiteware is further classified as earthenware, stoneware, chinaware,	
	porcelain and technical ceramics.	
Earthenware	Glazed or unglazed non vitreous (porous) clay based ceramic ware.	
	Applications include kitchenware, ovenware, tableware and tile.	
Stoneware	Vitreous or semi vitreous ceramic ware of fine texture, made primarily	
	from non refractory fire clay or some combination of clays, fluxes and	
	silica that when fired has property similar to stoneware made from	
	fireclay. Applications include chemicalware, cookware, drainpipe,	
	kitchenware, tableware and tile.	
Chinaware	Vitreous ceramic ware of zero or low absorption after firing that is	
	used for non technical applications. Applications include artware,	
	ovenware, sanitaryware, and tableware.	
Porcelain	Glazed or unglazed vitreous ceramics ware used primarily for	
	technical purposes. Applications include artware, ball mill balls,	
	chemical ware, insulators and table ware.	
Technical	Vitreous ceramics whiteware used for such products as electrical	
ceramics	insulation or for chemical, mechanical, structural or thermal	
	applications.	

Table 1.1: The general classifications of traditional ceramics (ACerS, 1920)

1.2 Background

The American Society for Testing and Materials (ASTM, 2002) defines a ceramic article as "an article having a glazed or unglazed body of crystalline or partly crystalline structure, or of glass, which body is produced from essentially inorganic, non-metallic substances and either is formed from a molten mass which solidifies on cooling, or is *formed and simultaneously or subsequently matured by the action of the heat*. The word ceramic is derived from a Greek word 'keramos' meaning pottery. It is related to an old Sanskrit root meaning 'to burn' but primarily, the Greeks used the term to mean "burnt stuff" or "burned earth". Thus the word was used to refer to a product obtained through the action of fire upon earthy materials.

Archeologists have discovered the human-made ceramics that date back to at least 24,000 BC. These ceramics were found in the form of animal and human figurines, slabs, and balls in Czechoslovakia. These ceramics were made of animal fat and bone mixed with bone ash and a fine claylike material. These ceramics were formed and then fired at temperatures in the range of 500 to 800°C in domed and horseshoe shaped kilns partially dug into the ground with loess walls. The function of these ceramics were not clear but is believed not to be the utilitarian one. The first use of functional pottery vessels is thought to be in 9,000 BC or 10,000 BC. These vessels were most likely used to hold and store water, grain and other foods. Tiles were manufactured in Mesopotamia and India almost 10,000 years later as settled communities had been established. Clay bricks were also made around the same time. Glass however was believed to be discovered in Egypt around 8,000 BC, when overheating of kilns produced a colored glaze on the pottery. Experts estimate that glass was produced independently of ceramics and fashioned into separate items before 1,500 BC.

Fast forward to the Middle Ages, the metal industry was in its infancy. At the time, furnaces were constructed of natural materials and were used for melting the metal. The industrial revolution was born when synthetic materials with better resistance to high temperatures called refractories were developed in the 16th century, These refractories created the necessary conditions for melting metals and glass on an industrial scale, as well as for the manufacture of coke, cement, chemicals, and ceramics.

Another major development occurred in the second half of the 19th century, when ceramic materials for electrical insulation were developed (ACerS, 1920). As other

inventions came on the scene including automobiles, radios, televisions and computers, ceramic and glass materials were needed to help these become a reality, as shown in the following timeline in **Table 1.2**.

Year	Development
24,000 BC	Ceramic figurines used for ceremonial
	purposes
14,000 BC	First tiles made in Mesopotamia and India
9,000-10,000 BC	Pottery making begins
5,000-8,000 BC	Glazes discovered in Egypt
1,500 BC	Glass objects first made
1550 AD	Synthetic refractories (temperature resistant)
	for furnaces used to make steel, glass,
	ceramics, cement
Mid 1800s	Porcelain electrical insulation
	Incandescent light bulb
1920s	High-strength quartz-enriched porcelain for
	insulators
	Alumina sparkplugs
	Glass windows for automobiles
1930s	-
1940s	Capacitors and magnetic ferrites
1950s	-
1960s	Alumina insulators for voltages over 220 kV
	Application of carbides and nitrides
1970s	Introduction of high-performance cellular
	ceramic substrates for catalytic converters and
	particulate filters for diesel engines
1980s	High temperature superconductors

 Table 1.2: Timeline of selected ceramic and glass developments (ACerS, 1920)

1.3 Problem Statement

Many forming processes used in ceramics such as dry pressing, extrusion, plastic forming and injection moulding are similar in principle of processing plastics and metals. However, the abrasiveness, wear resistance and hardness of ceramics are the difficulties faced in ceramic processing in order to avoid wear and contamination. Due to that problem, slip casting is chosen as a more specifically forming method used for ceramic processing. Good material homogeneity can also be achieved in slip casting. Furthermore, slip casting is a conventional method and the inexpensive mould material makes slip casting as a low cost fabrication method. In this research, ceramics to be fabricated is the porcelain ceramics with varying proportions of quartz and kaolin and it is sintered as high as 1250°C. Sintering temperature causes densification that gives the ceramic products strength and other properties that will be characterized in this research.

1.4 Objectives

The objectives of this research are:

- a) To produce high density sintered porcelain at sintering temperature of 1250°C.
- b) To identify the optimum composition of quartz and kaolin in yielding the highest bending strength of a porcelain body.
- c) To characterize and determine the mechanical strength of final product by varying the proportions of quartz and kaolin.

1.5 Scope

In this research, the study will be focused on the ceramic processing technique as a method to get high density porcelain ceramics when sintered at 1250°C. The kaolin and quartz proportions will be varied while other raw materials like feldspar, ball clay and

alumina will be kept constant. These raw materials will be mixed together using wet ball milling and cast into solid. The effects of varying proportions of kaolin and quartz on porcelain strength will be tested by drying and shrinkage test, flexural test, hardness test and tensile test. The morphology of the sintered porcelain body will be observed using SEM and XRD. The information getting from conducting the test will be compared so that the optimum compositions of kaolin and quartz required to get highest strength of the porcelain body can be achieved. Chapter One introduces the ceramics generally and its background history. Chapter Two describes mainly the porcelain and the previous research related to this research and Chapter Three describes the method of sample preparation and evaluation of final product. Chapter Four states the result and discussions and Chapter Five concludes the present research.

