The Effects of Chemical Treatment in the Traditional Creation of Peninsular *Keris* Blade

By

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Dissertation submitted in partial fulfillment of the requirements for the Bachelor of Engineering (Hons) (Chemical Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the Chemical Engineering Programme Universiti Teknologi PETRONAS in partial fulfillment of the requirement for the BACHELOR OF ENGINEERING (Hons) (CHEMICAL ENGINEERING)

Approved by,

(Assoc. Prof. Dr. Bambang Ari Wahjoedi)

UNIVERSITI TEKNOLOGI PETRONAS TRONOH, PERAK July 2010

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MOHD NORSHAHRIL JAILANI

ABSTRACT

The aim of this project is to investigate the effect of traditional chemical treatment used towards the properties of the keris blade, which are the strength, surface properties and chemical elements. According to Gardner (1936), the chemical treatments used are salt solution (Sodium Chloride), Sulphur powder, rice water and limejuice. The experiment is scoped for the chemical analysis towards the keris sample, not including the built of the keris or elemental distribution of the material. The keris sample had undergone chemical treatment in sequence, and at each stages of the treatment, the chemical analysis is performed, which are the Rockwell HRC Hardness Test, X-Ray Diffractometry Test (XRD), Field Emission Scanning Electron Microscopy / Energy Dispersive X-ray Test (FESEM/EDX), and Fourier-Transformed Infrared Spectroscopy Test (FTIR). Each analysis will determine different properties of the sample and data comparison can be made. The strength of the *keris* blade reduced after 2 stages of chemical treatment, but increased back after completion. Rust formation occurred in all four stages, while ferrous sulfide or Pyrite formed in stage 2. Stage 4 has the least corrosion due to acidic reaction and antioxidant properties of limejuice and creation of metal coating on surface. The removal of metal oxides had deteriorated the surface structure. In conclusion, the traditional chemical treatments used are proven scientifically using the latest methods of chemical analysis.

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CHAPTER 1 INTRODUCTION

1.1 Background

The *keris* is undeniably the most distinctive Malay weapon. Dated back from the reign of *Majapahit* to the glorious Malaccan Empire until now, used as weapon, mark of royal position and also as an art. The *keris* nowadays are considered as treasured heritage and highly prized among weapon collectors in Malaysia and from around the world.

The most essential part of *keris* is the *keris* blade. The blade is made of iron, but later perfected by infusing different types of metal like nickel, silver, cooper and, sometimes but rarely, gold, which then produced damascened *keris* or *Keris berpamor* (*keris* blade with damascened pattern).

The art of infusing and mixing irons with other metals had produced *keris* with significant strength, good balance and light-weighted, and this method is proven as to create a good *keris*. Other important method is the chemical treatment used by the *pandai besi* during and after the creation of single *keris*. The chemical treatment is used to further strengthen the blade and protects it from corrosion and defects, while enhancing the damascened pattern of the *keris*.

There are quite numbers of academic literatures and documentations explaining the scientific and technical description of damascened *keris*. However, there are not many literatures covering the scientific and technical aspect of the chemical treatment methods used for the *keris*, and only few of them are viable. Thus, documentation has to be done regarding the scientific and technical aspect of the chemical treatment process and its effects towards the traditional *keris* blade.

This project implemented and utilized the latest technology in chemical analysis in order to investigate the effect of chemical treatment towards the *keris*. The effects of the chemical treatments are investigated in four bases: the strength of the *keris*, its surface structure, its surface texture and surface chemical element(s), and its chemical group. The testing used are Fourier-Transformed Infrared Spectroscopy (FTIR) testing, Field Emission Scanning Electron Microscopy/Energy Dispersive X-Ray (FESEM/EDX) testing, X-Ray Diffractometry (XRD) testing and also Rockwell HRC Hardness Testing.

1.2 Problem Statement

1.2.1 Problem Identification

The method of infusing different metals together in creating *keris* blade is proven to strengthen the *keris*, but it is unknown whether the chemical treatment used could give the same effect towards the creation of the *keris*. Thus it is to be proven that the chemical treatment used is affecting the physical properties of the traditional *keris*.

The sample of *keris* that has been treated with traditional method of chemical treatment is analyzed using instrumental methods of Chemical analysis. The parameters for the analysis are the strength, surface structure, surface texture and chemical elements, and chemical groups of the *keris* sample.

1.2.2 Significance of Project

After doing a thorough research for documentations and articles related to *keris*, it is found that the traditional and current practice of chemical treatment towards *keris* is not well documented. And by completing this project, the current practice of the chemical treatment can be documented in a more systematic and technical method. The proper documentation of skills and methods of chemical treatment and *keris* making can be preserved, thus this project is significant as it subsequently purposed to preserve the *Keris* as part of Malay heritages.

1.3 Objectives and Scope of Study

The main objective of this project is to study the effect(s) of chemical treatment in the traditional creation of peninsular *keris* blade using the latest technology of chemical analysis methods and according to the analysis parameters. Other objectives would also to study the reasons behind the usage and applications of the traditional treatment on *keris* by the old folklore.

The project is scoped only for the chemical treatment and analysis towards the sample of *keris*, not including the built of the *keris*, the elemental distribution of the *keris* blade, the artistic designs of the blade, hilt or sheath, or the cultural and spiritual view of a certain culture towards *keris* as a weapon.

1.4 Relevancy of Project

This project is relevant to my engineering background generally, and to my discipline of Chemical Engineering specifically, because this project involve a thorough scientific research and technical skills as this project requires good fundamental and advanced chemical analysis method, using advanced technological instrumentations and also good laboratory conducts.

1.5 Feasibility of Project

This project is feasible to be executed and completed because the tools required to run the experiment is available in Chemical Engineering Laboratory building, which is the FTIR-8400S Fourier-Transformed Infrared Spectrophotometer (OEM: Shimadzu). Other than that, the equipments for other testing, such as FESEM/EDX, XRD and Hardness test also available in the Material Laboratory building, Mechanical Engineering Department (building 17). The timeline of this project is estimated to fit the 2 semester's period, so the project can be completed within the estimated time period.

CHAPTER 2

LITERATURE REVIEWS AND/OR THEORIES

2.1 The built of Keris Blade

Keris is one of the traditional Malay weapons, including spear, sword and dagger. Considered to be a weapon long ago, now *keris* is considered as art and a heritage that needs to be preserved. Nowadays, there are not many *pandai besi* or someone with the qualified skills and knowledge to create *keris*. Thus it is very important to record and document all the skills and methods in *keris* making for future reference. (Gardner,1936; Yuwono, 2008)



Figure 2.1: the traditional keris berpamor or damascened keris with sheath.

G.B. Gardner(1936), in his book '*Keris* and Other Malay Weapons' said that *keris* is primarily a dagger with an angled hilt, sort of like pistol grip that enable the wielder to thrust at greater reach, although the thrust lacks the force from the swing of weapon such as sword. It also categorized as rapier.

According to Malay tradition, a *keris* must be made of at least two kinds of metals. The methods of infusing and layering two or more different types of metals or irons are perfected over time, which then produced *keris berpamor* or damascened *keris* (*keris* with Damascus patterns). The damascened *keris* blade is said to be stronger and lighter compared to those made with pure iron. (Gupta, 2009) Ever since then, the *keris* blade is created with the Damascus technique. (Gardner, 1936)

2.2 The traditional chemical treatment methods

One of the elements of *keris* making is the chemical treatment done towards the keris. The purpose of the chemical treatment is said to strengthen the bond between metals in the *keris* and to make the Damascus pattern more vivid and beautiful.

According to G. B. Gardner (1936), the tradition method of the chemical treatment used is by soaking the *keris* in a through containing salt, sulfur and boiling rice water for three or four days. "This blackens the steel but scarcely touches the iron. It attacks the marks of the welds, which shows as tiny etched lines," claimed Gardner. The sequential chemical treatment methods make the Damascus pattern more vivid, and when the damascened pattern is clear, the *keris* blade is cleansed using limejuice. Thus, by referring to Gardner's explanation of the traditional chemical treatment method, the chemical substances used are:

- Salt solution, Sodium Chloride (NaCl).
- Sulfur powder.
- Rice water at boiling temperature.
- Extract of limejuice, containing citric acid.

According to some other Malay tradition, the *keris* is soaked in coconut water instead of rice water. There is also other common tradition of coating the *keris* blade with natural poison such as stingray poison and arsenic (Gardner, 1936).By knowing the chemical substances and solutions, the chemical analysis can be implemented towards the sample.

2.3 The Traditional Chemical Solutions / Substances Used

2.3.1 Salt solution

The first solution used for the traditional treatment towards *keris* is salt. The type of salt probably used is the common table salt, or Sodium Chloride, with the formula NaCl. Sodium Chloride is an ionic compounds that usually in the form of crystal powder, and able to dissolve in water with solubility of 360g NaCl / kg H₂O at 25°C.(Feldman, 2005) The reason behind the usage of common salt is that it was widely used during the ancient time as food flavoring, as cleaning agent and also it is used as part of the tradition ritual.

Since that the *keris* was known as a weapon for combat, there are possibilities that it caused cutting and stabbing to human body, thus leaving it in bloody condition. Thus the salt is used to clean the blood on the blade, either by scrubbing the blade with salt powder or soaking it in salt solution. For the treatment method, the *keris* sample is soaked inside the salt solution over a period of time. However, prolonged treatment may lead to ferrous corrosion depending on the substances of the *keris* sample and corrosion rate. (Lin & Wang, 2005)

2.3.2 Sulfur Powder

Sulfur or sulphur is an abundant, multivalent non-metal chemical element, with formula S. Its native form is a bright yellow crystalline solid. In nature, it can be found as pure element and/or as sulfide and sulfate minerals. It able to dissolve in many solvents, including water, which will produce the corrosive Hydrogen Sulfide, H₂S. Sulfur is also corrosive to most metals, except copper and copper-alloy. The sulfur powder is used in the chemical treatment of *keris* to remove rust and to make the blade darker. The prolonged reaction between iron and sulfur produces Ferrous Sulfide, also known as pyrite.

According to Adel L. Pfeil (1990):

Hydrogen sulfide dissolved in water corrodes metals such as iron, steel, copper and brass. The corrosion of iron and steel from sulfur forms ferrous sulfide or "black water." Hydrogen sulfide in water can blacken silverware and discolor copper and brass utensils.

2.3.3 Rice Water

Rice water is obtained through the washing of the rice during the cooking process. It also mainly obtained from the froth produced on the surface of boiling rice. From the literature stated by Gardner (1936), the boiling rice water probably referred to the froth or the water mixed with the rice while cooking it. The content of the rice water is rich with starch, a naturally abundant nutrient carbohydrate, $(C_6H_{10}O_5)_n$. (Wong,1981)

The starch components might have been extracted from the rice grain while being cooked. The starch is found chiefly in the seeds, fruits, tubers, roots, and stem pith of plants, notably in corn, potatoes, wheat, and rice, and varying widely in appearance according to source but commonly prepared as a white amorphous tasteless powder. (Answer, 2010). Asian women famously use the rice water as a source of beauty when used to wash the face or take a bath. (Wikipedia, 2010). The rice water is used to clean the blade from rust and sulfur residue from previous treatments.

2.3.4 Limejuice

The last component used in the traditional chemical treatment towards *keris* is the limejuice. The limejuice is extracted as juice from the citrus fruit primarily limes fruit. The types of lime commonly used are key lime (*Citrus aurantifolia*) locally known as '*limau nipis*', and also grapefruit (*Citrus Medica*) locally known as '*limau purut*'. There are also other types of lime being used, depending on the concentration and quantity required for cleaning.

The limejuice contains high concentration of citric acid and ascorbic acid, which is Vitamin C. it also high on sugar and water content and acts as antioxidant. It is traditionally used as a cleaning agent, and in the case of *keris* cleaning; it acts as an antibacterial coating on the blade. Besides, it is claimed that the lime is used traditionally to chase away evil spirits. (Arias & Ramon-Luca, 2004)

2.4 Instrumental Methods of Chemical Analysis

The effects of the traditional treatment are measured according to the parameters using the high-tech analytical equipment used for Non-Destructive Test (NDT) (Rouessac&Rouessac, 2007), as follows:

- 1. The strength of the *keris* sample
- 2. The surface structure of the keris blade
- 3. The surface texture image of the keris blade
- 4. The surface chemical elements and groups existed on the keris blade

Each of the properties is investigated using different kinds of analytical equipments, thus the results of the analysis is estimated to cover various aspects of the properties of the *keris*. All of the testing must be done in the same stage, after treated with the same solution and according to the testing sequence in order to establish relations between the results.

2.4.1 Fourier-Transformed Infrared Spectroscopy (FTIR)

Fourier Transform-Infrared Spectroscopy (FTIR) is an analytical technique used to identify organic (and in some cases inorganic) materials. This technique measures the absorption of infrared radiation by the sample material versus wavelength. The infrared absorption bands identify molecular components and structures.(Rouessac&Rouessac, 2007)

When a material is irradiated with infrared radiation, absorbed IR radiation usually excites molecules into a higher vibrational state. The wavelength of light absorbed by a particular molecule is a function of the energy difference between the atrest and excited vibrational states. The wavelengths that are absorbed by the sample are characteristic of its molecular structure.

The FTIR spectrometer uses an interferometer to modulate the wavelength from a broadband infrared source. A detector measures the intensity of transmitted or reflected light as a function of its wavelength. The signal obtained from the detector is an interferogram, which must be analyzed with a computer using Fourier transforms to obtain a single-beam infrared spectrum. The FTIR spectra are usually presented as plots of intensity versus wave number (in cm⁻¹). Wave number is the reciprocal of the wavelength. The intensity can be plotted as the percentage of light transmittance or absorbance at each wave number.

(Materials Evaluation and Engineering, Inc, 2009)

2.4.2 Field Emission Scanning Electron Microscopy (FESEM)

Scanning electron microscopy (SEM) or Field Emission Scanning Electron Microscopy (FESEM) is a method for high-resolution imaging of surfaces. The SEM uses electrons for imaging, much as a light microscope uses visible light. The advantages of SEM over light microscopy include much higher magnification (>100,000X) and greater depth of field up to 100 times that of light microscopy. Qualitative and quantitative chemical analysis information is also obtained using an energy dispersive x-ray spectrometer (EDS) with the SEM. (West, 1999; Ewing, 1985)

The SEM generates a beam of incident electrons in an electron column above the sample chamber. The electrons are produced by a thermal emission source, such as a heated tungsten filament, or by a field emission cathode. The energy of the incident electrons can be as low as 100 eV or as high as 30 keV depending on the evaluation objectives. The electrons are focused into a small beam by a series of electromagnetic lenses in the SEM column. Scanning coils near the end of the column direct and position the focused beam onto the sample surface. The electron beam is scanned in a raster pattern over the surface for imaging. The beam can also be focused at a single point or scanned along a line for x-ray analysis. The beam can be focused to a final probe diameter as small as about 10 Å.

(Materials Evaluation and Engineering, Inc, 2009)

2.4.3 X-Ray Diffractometry test (XRD)

X-ray diffractometry (XRD) is a versatile, non-destructive technique that reveals detailed information about the chemical composition and crystallographic structure of natural and manufactured materials. (Rouessac & Rouessac, 2007) A crystal lattice is a regular three-dimensional distribution (cubic, rhombic, etc.) of atoms in space. These are

arranged so that they form a series of parallel planes separated from one another by a distance d, which varies according to the nature of the material. For any crystal, planes exist in a number of different orientations - each with its own specific d-spacing.

When a monochromatic X-ray beam with wavelength *lambda* is projected onto a crystalline material at an angle *theta*, diffraction occurs only when the distance traveled by the rays reflected from successive planes differs by a complete number n of wavelengths.

By varying the angle *theta*, the Bragg's Law conditions are satisfied by different *d*-spacings in polycrystalline materials. Plotting the angular positions and intensities of the resultant diffracted peaks of radiation produces a pattern, which is characteristic of the sample. Where a mixture of different phases is present, the resultant diffractogram is formed by addition of the individual patterns.

Based on the principle of X-ray diffraction, a wealth of structural, physical and chemical information about the *keris* can be obtained. A host of application techniques for various material classes is available, each revealing its own specific details of the sample studied. (PANalytical, 2010)

2.4.4 X-Ray Fluorescence Spectrometry test (XRF)

X-ray fluorescence spectrometry (XRF) is a non-destructive analytical technique used to identify and determine the concentrations of elements present in solid, powdered and liquid samples. XRF is capable of measuring elements from beryllium (Be) to uranium (U) and beyond at trace levels often below one part per million and up to 100%.



Figure 2.2: The XRF spectrometer measures the individual component wavelengths of the fluorescent emission produced by a sample when irradiated with X-rays.

WDXRF (wavelength dispersive X-ray fluorescence) separation is achieved by diffraction, using an analyzer crystal that acts as a grid. The specific lattice of the crystal selects the correct wavelengths according to Bragg's Law. A WDXRF spectrometer provides:

- The advantages of total application versatility
- Optimal measurement conditions programmable for each element
- Excellent light-element performance
- Very high sensitivity and low detection limits.

EDXRF (energy dispersive X-ray fluorescence) spectrometry works without a crystal. An EDXRF spectrometer includes special electronics and software modules to take care that all radiation is properly analyzed in the detector. It provides a lower cost alternative for applications where less precision is required. The high-end Epsilon 5 XRF spectrometer uses the 3D EDXRF techniques featuring a 3-dimensional, polarizing optical geometry. (PANalytical, 2010)

2.4.5 Hardness Testing

Hardness is the property of a material that enables it to resist plastic deformation, usually by penetration. However, the term hardness may also refer to resistance to bending, scratching, abrasion or cutting. Hardness is not an intrinsic material property dictated by precise definitions in terms of fundamental units of mass, length and time. A hardness property value is the result of a defined measurement procedure. There are three principal standard test methods for expressing the relationship between hardness and the size of the impression, these being Brinell, Vickers, and Rockwell. For practical and calibration reasons, each of these methods is divided into a range of scales, defined by a combination of applied load and indenter geometry. (England, 2010)

The Rockwell hardness test method consists of indenting the test material with a diamond cone or hardened steel ball indenter. The indenter is forced into the test material under a preliminary minor load *F0* usually 10 kgf. When equilibrium has been reached,

an indicating device, which follows the movements of the indenter and so responds to changes in depth of penetration of the indenter is set to a datum position. While the preliminary minor load is still applied an additional major load is applied with resulting increase in penetration. When equilibrium has again been reach, the additional major load is removed but the preliminary minor load is still maintained. Removal of the additional major load allows a partial recovery, so reducing the depth of penetration. The permanent increase in depth of penetration, resulting from the application and removal of the additional major load is used to calculate the Rockwell hardness number.(England, 2010)

$\mathrm{HR} = E - e$

Where, F0 = preliminary minor load (kgf)

- F1 = additional major load (kgf)
- F = total load (kgf)
- e = permanent increase in depth of penetration due to major load F1 measured in units of 0.002 mm
- E = a constant depending on form of indenter: 100 units for diamond indenter, 130 units for steel ball indenter

HR = Rockwell hardness number

D =diameter of steel ball



Figure 2.3: Rockwell Principle

Seele	Indonton	Minor Load,	Major Load,	Total Load,	Value of
Scale	muenter	<i>F0</i> (kgf)	F1 (kgf)	F (kgf)	E
А	Diamond cone	10	50	60	100
В	1/16" steel ball	10	90	100	130
С	Diamond cone	10	140	150	100
D	Diamond cone	10	90	100	100
Е	1/8" steel ball	10	90	100	130
F	1/16" steel ball	10	50	60	130
G	1/16" steel ball	10	140	150	130
Н	1/8" steel ball	10	50	60	130
K	1/8" steel ball	10	140	150	130
L	1/4" steel ball	10	50	60	130
М	1/4" steel ball	10	90	100	130
Р	1/4" steel ball	10	140	150	130
R	1/2" steel ball	10	50	60	130
S	1/2" steel ball	10	90	100	130
V	1/2" steel ball	10	140	150	130

Table 2.4.5: Rockwell Hardness scale

HRA — Cemented carbides, thin steel and shallow case hardened steel

HRB — Copper alloys, soft steels, aluminium alloys, malleable irons, etc.

HRC — Steel, hard cast irons, case hardened steel and other materials harder than 100 HRB

- HRD Thin steel and medium case hardened steel and pearlitic malleable iron
- HRE Cast iron, aluminium and magnesium alloys, bearing metals
- HRF Annealed copper alloys, thin soft sheet metals
- HRG Phosphor bronze, beryllium copper, malleable irons
- HRH Aluminium, zinc, leads
- HRK
- HRL
- HRM HRP

Soft bearing metals, plastics and other very soft materials

HRS

HRR

HRV

CHAPTER 3 METHODOLOGY

3.1 Research Methodology

The literatures and articles regarding the *keris* is obtained online, mostly indicates the general forms and history of *keris*. The book *Keris*& Other Malay Weapons (Gardner, 1936) is considered the main reference regarding the *keris* as it has extensive and detailed informations about the history and the element of *keris*.

The book Instrumental Methods of Chemical Analysis by Galen W.Ewing (1985) and Chemical Analysis: Modern Instrumentation Methods and Techniques (2007) is used as a reference and guidelines in the chemical analysis process used in testing the sample. Other than that, there are journal articles and literatures that explain generally about particular chemical analysis.

3.2 **Project Methodology**

This project is experimental as the result of each chemical analysis is unknown. The sample (*keris*) is treated with the traditional methods of chemical treatment one at a time, then proceeded with chemical analysis. The chemical analysis is peformed in each steps to study the effects of sequential treatment. Once the sample is completely treated with every said chemical accordingly, the final effects can be detected and analysed.

Based on the test results, the acquired data then can be used to determine the real chemical substances that actually reacted or causing changes towards the sample, regardless if it actually improving or deterioriating the properties of the *keris*. And by determining the substances, it can be extracted from the traditional source or synthesized thus can be used effectively towards the treatment process.

3.2.1 Pre-testing of *Keris* sample

The *keris* sample is subjected for a preliminary testing of all the chemical analysis in order to set the preliminary standards and as a benchmark point for result comparisons. The sample will undergo the following testing:

- 1. Hardness testing, using Rockwell HRC Hardness Tester.
- 2. X-Ray Diffractometry (XRD) testing, using X-Ray Diffraction Sprectrometer.
- Field Emission Scanning Electron Microscopy (FESEM)/Energy Dispersive X-ray (EDX) testing, using Electron Microscope.
- 4. Fourier-Transformed Infra Red (FTIR) testing, using Fourier-Transformed Infraspectrometer.



Figure 3.1: the initial *keris* condition for pretesting.

The important precaution of this pre-testing is that the *keris* sample must not be treated with any kinds of chemicals or solutions, other than distilled water. The sample must be in clean condition to achieve the pre-treatment results. The *keris* sample is then cut into two major parts and two minor parts to be used in the analysis. Once the results are obtained, the data can be used as the initial data before proceeding to the chemical treatment.

3.2.2 Preparation of the Chemical Solutions

The solutions and mixtures used for the chemical treatment are prepared at specified amount, concentration and specification that will closely resemble the tradtional ways of preparation process. TheSodium Chloride solution is prepared by diluting 5g of solid Sodium Chloride, NaCl or commonly known as table salt in 400mL of water at room temperature. The Sulphur powder is acquired from laboratory, and no further preparation required. The safety rules must be obeyed while handling the powder, including wearing protective gloves and wearing goggles.



Figure 3.2: prepearation of salt solution.

For the rice water, the froth is prepared by boiling the water containing rice. The method is similar to preparing and cooking the rice. The froth is skimmed of the surface and the water is collected from the pan. For the limejuice extract, the juice of the key lime is squeezed and extracted to produce high concentrate without diluting it.

The solutions can be prepared prior to the stage of the chemical analysis which the solution is used. The safety regulations must be obeyed while handling and preparing the solutions. Once the solution is completed, the chemical treatment can be performed onto the *keris* sample.

3.2.3 The chemical treatment methods

After preparation of the chemical solution or the respective stage, the *keris* sample is treated with the solution by soaking the sample completely into the solution. However, each solutions have different soaking period and conditions, such as:

- 1. Stage 1: treatment with salt solution:
 - The sample is immersed in the Sodium Chloride solution for 24 hours at room temperature, 25°C.
- 2. Stage 2: treatment with Sulphur:
 - The sample is powdered with the sulphur powder as a coating. The

treatment is to be done for only 6 hours, due to the nature of the sulphur that may cause mild corrosion to metals.

- 3. Stage 3: treatment with rice water:
 - The sample is soaked in the water bath containing rice water for 24 hours at room temperature, 25^oC.
- 4. Stage 4: treatment with limejuice:
 - The sample is soaked in the limejuice extract at room temperature for 24 hours period.



Figure 3.3: powdering of keris sample with sulfur.

3.2.4 The Chemical Analysis Methods

Once the *keris* sample has reach the soaking time of the respective stage, the *keris* sample is taken out from the solution. The sample is air-dried from the solution without wiping to avoid any changes from treatment effects. After that, the chemical analysis methods are performed towards the *keris* sample to determine any effects or changes in terms of the properties on the sample:

- 1. Hardness Testing
- 2. XRD testing
- 3. SEM/EDX testing
- 4. FTIR testing

The analysis is performed three to five times and the average is obtained to produced accurate results. After completing the respective stage, the *keris* sample is then

prepared for the next stage of chemical treatment. It is reminded that, the sample must not be washed or cleaned before proceeding with the next chemical treatment. This is to create accumulative effects on the *keris* sample as it was performed by the folkslore. The next stage of chemical treatment is performed although there are traces of the previous chemical substance on the *keris* sample.

3.2.4.1 Chemical Analysis 1: Fourier-Transformed Infrared Spectroscopy Testing

After performing the pre-testing process, the sample proceed with the first stage of chemical treatment with salt solution. In this stage, the *keris* sample is immersed in the Sodium Chloride solution for the period of 24 hours at room temperature, 25° C. After the first stage, it is found that the solution contains traces of yellow-orange particles, presumably the rust from the sample. The sample is taken out from the solution and left dry. The sample is filed to obtain metal powder to be used for the testing. After the testing, the result of the sample is produced in terms of graphical plot of percentage transmittance (%T) versus absorption peak (1/cm).

(Materials Evaluation and Engineering, Inc, 2009)

For the second stage, the sample undergo chemical treatment with sulphur powder. The *keris* sample is coated with sulphur powder for surface reactions. The treatment is performed for 6 hours, due to the corrosive nature of sulphur towards metal. (Pfeil,1990) After the second stage, the sample is washed off of the powder and let dry. From the treatment, traces of dark lines are visible on the layers of the *keris* sample. The sample is filed for the FTIR testing and the result is obtained.

For the third stage, the *keris* sample undergo chemical treatment with rice water. The sample is immersed inside a container with the rice water at room temperature for 24 hours. After the treatment, the sample is taken out and let dry. The sample is filed for the FTIR testing and the result is obtained.

For the fourth stage, the sample undergo chemical treatment with limejuice. The *keris* sample is immersed inside a container with key lime extract at room temperature for 24 hours. After the treatment, the sample is taken out and let dry. The sample is then filed for the FTIR testing and the result is obtained.

3.2.5 Data Acquisition methods

The results can be obtained after the testings, and this ease up the data intepretation and comparison process. Each data is sorted in stage sequence to detect the changes occurred in the process. The changes is then investigated and discussed to obtain the results using technical and specific methods.

Testings	Reading, R									
	R1	R2	R3	R4	R5	R				
Strength of keris										
Chemical elements										
Surface texture										
Surface structure										

Table 3.2.5: Results on each stage

Table 3.2.6: Result comparisons

Testings	Reading from stages, S							
	1	2	3	4	5	Comments		
Strength								
Surface structure								
Surface texture								
Surface chemical elements								
Chemical groups								

3.2.6 Sample powdering (filing)

For the FTIR testing, the *keris* sample have to be filed to produce metal powder or dust and to be used for the equipments. The amount needed is very small, approximately 5 mg of the metal powder from the *keris* sample. The method used to obtain the powder is by applying rough file and grating onto the surface of the sample using metal filer.



3.3 Project Methodology (flowchart)

Figure 3.4: The process flowchart for the project methodology.

3.4 Project Planning

No.	Detail/week on semester 1	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of project topic														
2	Preliminary research work about <i>keris</i> .														
3	Acquisition of keris sample.														
4	Further research about chemical analysis.														
5	Submission of Progress Report.														
6	Seminar presentation														
7	Pretesting Analysis: SEM & XRD														
8	Pretesting Analysis: FTIR & HRC														
9	Submission of Interim Report Final Draft.														
10	Oral presentation														
11	Planning on Semester 2 activities														
No.	Detail/week on semester 2	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Planning for continuing project work.														
2	Chemical Analysis: FTIR														
3	Chemical Analysis: HRB														
4	Analysis and verification of Test results														
5	Submission of Progress report					_									
6	Preparing for Optimization testing.														
7	Chemical Analysis: SEM														
8	Chemical Analysis: XRD														
9	Submission of progress report 2.														
10	Poster design and printing														
11	Poster exhibition														
12	Project completion.														
13	Submission of Dissertation (soft bound)														
14	Oral presentation.														
15	Submission of Project Dissertation(hard bound)														

3.5 Tools Required

The sample of *keris* is used for the experimental process. As for the chemical analysis process, the instruments such as the spectrophotometer and XRD/XRF equipments are available in chemical engineering and mechanical engineering laboratory, respectively. The consumables can be acquired through the laboratory using the requisition form.



Figure 3.5: the equipments used for chemical analysis.

CHAPTER 4 RESULTS & DISCUSSIONS

4.1 Parameter: Strength

According to the methodology, the strength of the *keris* is measured by using the Rockwell HRC Harness testing. The type of indenter used is the diamond cone (E = 100 unit). The results of the hardness testing are as follows:

Stage of	Total load,		Hardness value (HRC)										
treatment	F (kgf)	R ₁	R ₂	R ₃	R ₄	R ₅	R ₆	Ravg					
Pretesting	150	98.7	98.2	98.9	98.9	98.1	98.5	98.55					
Stage 1	150	92.5	98.3	97.7	98.4	94.9	97.5	96.55					
Stage 2	150	96.9	95.0	90.2	93.8	91.7	95.3	93.82					
Stage 3	150	95.8	96.9	94.5	91.9	95.2	96.2	95.08					
Stage 4	150	95.8	98.9	98.6	98.7	98.9	98.5	98.23					

Table 4.1: Rockwell HRC hardness result in 6 iterations



Figure 4.1: Rockwell HRC Hardness results

The average hardness value before the treatment is 98.55 HRC. Then, after stage 1 and stage 2, the hardness value decreases to 93.82 HRC. This is due to deteriorations caused by Ferrous (II) Oxide, FeO formation in Stage 1 and Ferrous Sulfide, FeS₂ formation in Stage 2. The layer of rusts and mild layer of Pyrite deteriorate the surface structure and strength due to reactions with Sodium Chloride and Sulfur.

After stage 3 treatment using rice water, the hardness increases and after stage 4, the hardness value increases to almost its original condition, 98.23 HRC. At stage 4, the increased hardness value is probably due to metal and acid reaction between the blade surface and the limejuice which then produce fine metal salts precipitate. The metal salts fills in the pores between the metal bands and on the surface of the blade thus strengthen the blade. It also provides a thin layer of coating on the surface that acts to prevent corrosion or rusts on the blade.

4.2 **Parameter: Chemical Elements and Groups**

The testing of Field Emission Scanning Electron Microscopy (FESEM) and Energy Dispersive X-Ray Spectroscopy (EDX) is used for the analysis of the chemical elements and groups on the *keris* blade. For FESEM, two magnifications are used: 100X magnification and 1000X magnification. The following graphs are obtained from the EDX testing:



Figure 4.2: EDX result of *keris* pretesting.

For the pretesting of the *keris* sample, the EDX shows that the sample consists of iron (50.71 weight% at 6.22 keV), calcium, silicon, aluminum (0.55 weight%), and carbon other that metal oxides on the surface. The surface of the sample shows mild rusting, probably due to the previous coating still in effect. The compounds existed are metal oxides such as FeO, SiO₂, and traces of Al₂O₃.



Figure 4.3: EDX result for stage 1of keris treatment.

In stage 1, the existence of FeO due to oxidization of iron after reacted with Sodium Chloride. There is also mild formation of Al_2O_3 between the different metal bands. Also, an interesting finding of arsenic, As (1.45 wt%), proven that poison is also being used as one of the treatment methods by the old folklore. The arsenic is likely been used for combat, hinting that this *keris* once being used by a Malay warrior or someone with good martial arts background, especially *silat*.



Figure 4.4: EDX result for stage 2 of keris treatment.

In stage 2, the sulfur reacted with the metals such as iron and produced Ferrous sulfide, or commonly known as pyrite. This mild formation of pyrite gives a nice glint of golden traces at the edge of the metal bands, hence the reason why some people called it "Fool's Gold". The sulfur also caused dark coloration on the blade due to its property to blacken the color of most metals such as iron and silver and the different dark hues depending on types of metals. The old folklore used this treatment to enhance its characteristics and making it looked more attractive, as a way to invite other people to buy the *keris*.



Figure 4.5: EDX result for stage 3 of keris treatment.

In stage 3, the finding of Phosphorus (0.66 wt%) and Magnesium oxide, MgO (Mg = 1.21 wt%) after reaction with rice water occurred. This is probably caused by nutrient and mineral attachment from the starch solution to the metal. Simple bonding formed of phosphorus ions and magnesium towards the surface metals during the chemical treatment. Alas, no concrete justification for the stage 3 treatment to explain scientifically the effects of the rice water.



Figure 4.6: EDX result for stage 4 of keris treatment.

In stage 4, the amount of metal oxide is small compared to the stage 1, due to the ascorbic acid property as an antioxidant mechanism and became dehydroascorbic acid, proving that the limejuice treatment provide a thin coating on the blade to prevent further corrosion. The reaction of ascorbic acid and citric acid with the metals has produced small amount of metal salts.

4.3 **Parameter: Surface Structure**

Stage 1

Stage 2



Stage 3 Stage 4 Figure 4.7: 100X magnification on *keris* surface using FESEM

The *keris* blade is created using multiple layer of metals: iron, nickel, Aluminum and Molybdenum, which latter two found in small amount. The low magnification on the blade (100X) shows the three different layers on the near-tip of the blade. The carbon

traces found in the iron shows that the iron contains carbon impurities and classified as carbon steel. The alloy is produced during the heat treating process in the creation stage, making it harder and stronger but less ductile.



Figure 4.8: XRD result for stage 2 of keris treatment.

The iron compound is the dominant element in the *keris* structure. Throughout the chemical treatment process, the *keris* sample exhibit small changes in structure, especially on the surface. The rust and metal oxides formation on the surface had reduced its thickness upon removal.

4.4 Parameter: Surface Texture

The chemical treatments performed on the blade left visible effect on its surface texture. In stage 1, the rust formation on the surface damaged the texture of the metal bands and layering, only to be then removed in stage 2 with sulfur. The rust and the ferrous sulfide layers completely covered the whole surface. The sulfur reaction with the metal had caused dark coloration different metals on the blade, thus create more vivid linings between different metal bands.

In stage 3, the treatment with rice water cleanses the blade from sulfur and rust residue. Then in stage 4, the limejuice cleanses the different dark coloration and brightens the Nickel on the blade, thus creating more vivid and visible layering between dark and light metal bands. After completing the treatment, the *keris* blade become more fine-looking with distinct layering of different metal bands.



Figure 4.9: 1000X magnification on keris surface using FESEM.

4.5 DISCUSSION: Comparison of results with traditional understandings

After completed the FESEM/EDX, Hardness testing, FTIR testing and XRD testing, it is found that there are similar comparisons between the traditional understandings of chemical treatment with the scientific experimental results.

Traditional explanation:

According to the old Malay sayings, the salt is used to cleanse the blade from blood after a combat or battle. The sulfur is used to blacken the blade and if the *keris* is used in a combat performance for royalty, the sulfur will create sparks when clashed with the opponent's *keris* blade and making the performance more

attractive. As for the rice water, the old '*pantang*' claimed that the blade is soaked in rice water in order to calm the blade from causing unintentional ruckus and massacre which may claimed innocent lives. The limejuice is used to purify the blade and to chase away evil spirit. All these *pantang* provide the mystical and common explanations towards the usage of the chemical treatment, and none of them explain scientifically the effects of the treatment.

Scientific explanation:

Based on the experimental works done on the *keris* blade, the usage of the salt cleanse the surface and subsequently leads to rust formation, which then when eliminated, the rust will also remove the dirt and unwanted substances along with it. Formation of Ferrous Sulfide creates golden linings on the metal bands, making it more attractive, while the sulfur blackens the metals to create vivid coloration between different layers. Fire sparks may also be created upon forced contact with other metals. The rice water washes the blade from the rust and sulfur residue. The limejuice used provides bright coating on the surface as a way to avoid corrosion on the blade.

By comparing both explanations, if is found that the usage of salt, sulfur and limejuice are able to be justified scientifically by using latest methods of chemical analysis. The old folklore has applied this practice for centuries probably without knowing clearly the scientific justifications toward the chemical treatment practices. For the rice water treatment, the traditional understanding is solely based on spiritual aspect of the treatment, thus no concrete scientific justification for the rice water treatment.

CHAPTER 5 CONCLUSION & RECOMMENDATIONS

5.1 Conclusion

In conclusion, the objective of the project is achieved, that is to study the effects of the chemical treatment towards the traditional *keris* blade according to the defined parameter. Based on the results, the chemical treatment is proven to strengthen the blade, create vivid coloration on the Damascus pattern on the *keris* surface and prevent corrosion on the blade.

5.2 **Recommendation**

After completing the research, there are some issues arises during the experimental works and discussion. Because of that, a few recommendations for the future works will provide better concept to solve the issues and to obtain solutions.

- The chemical treatments being used can be further defined in terms of concentration, temperature of solution, acidity and period of treatment to obtain more optimum effects of chemical treatment.
- Instead of using the traditional methods of chemical treatment, a better alternatives can be suggested and be used, such as using specific type of acid solution to achieve the desired effect of the chemical treatment. The work however must state the modern implementation of the chemicals toward the *keris*.
- A better finishing can be applied towards the *keris* surface, such as using metal epoxy or varnish as metal coating to prevent total corrosion on the *keris* surface.

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APPENDICES

KERIS SAMPLE A)



Figure A-1: the keris sample before cleaning (left) and after cleaning (right).



Figure A-2: the *keris* sample is cut into 4 pieces.

CHEMICAL ANALYSIS 1: FTIR Testing B)



Figure B-1: Stage 1 and Stage 2 of FTIR testing.

C) **RESULTS**

Figure C-1: Infraspectroscopy peak graph plot of the FTIR testing for Stage 1.

Figure C-2: Infraspectroscopy peak graph plot of the FTIR testing for Stage 2.

Figure C-3: Infraspectroscopy peak graph plot of the FTIR testing for Stage 3.

Figure C-4: Infraspectroscopy peak graph plot of the FTIR testing for Stage 4.

D) **RESULTS**



X-Ray Diffractometry peak graph of the XRD testing