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LIST OF SYMBOLS

ε	$-$	Small non-Archimedean number (10^{-6})
λ_j	$-$	unknown weight
u_r	$-$	Envelopment output weight
v_i	$-$	Envelopment input weight
V_i	$-$	Multiplier input weight
μ_r	$-$	Multiplier output weight
α	$-$	Proportion of input increment
γ	$-$	Proportion of output increment
β_1	$-$	Frontier shift in Period 1
β_2	$-$	Frontier shift in Period 2
θ	$-$	Input oriented efficiency score
Θ	$-$	Input/Output mix
s	$-$	Number of outputs
m	$-$	Number of inputs
DMU_0	$-$	Targeted DMU
DMU_j	$-$	Number of DMU in universal set n
z_0	$-$	Error measurement of DMU_0
x_{i0}	$-$	Unit of ith input consumed by DMU_0
y_{r0}	$-$	Unit of rth output produced by DMU_0
x_{ij}	$-$	Unit of ith input consumed by all DMUs include DMU_0
Φ	$-$	Pairwise comparison matrix
a_i	$-$	i^{th} decision alternatives
$\overline{W}$	$-$	Average vector
$\overline{w}$	$-$	Weights vectors

δ_{max}	–	Eigenvectors
y_{rj}	–	Unit of rth output produced by all DMUs including DMU_0
s_r^+, s^+	–	Output deficit
s_i^-, s^-	–	Input surplus
$w_i{}^-$	–	Input user specified weights
$w_r{}^+$	–	Output user specified weights
A_i	–	Output weight
B_r	–	Transformed input weight
η_i	–	Input weight
ξ_r	–	Transformed output weight
θ^{super}	–	Input oriented super efficiency score
ϕ^{super}	–	Output oriented super efficiency score
e_r	–	Ratio of output increment
I	–	Universal set of input variable
O	–	Universal set of output variable
I_D	–	Subset of input discretionary variables
O_D	–	Subset of output discretionary variables
I_N	–	Subset of input non discretionary variables
O_N	–	Subset of output non discretionary variables
L	–	Set of DMUs into categories
p_i^0	–	i^{th} input price for DMU_0
$\tilde{x_{i0}}$	–	i^{th} input that minimizes cost
χ^2	–	Chi-Square Statistic
r_{s_c}	–	Spearman's Corrected Rho
T	–	Number of Ties
ξ_h	–	Ratio of output increment
z_h	–	Normally distributed
v_h	–	Ordinarily exponential
σ^2	–	Variance
H_0	–	Null Hypothesis
H_1	–	Alternative Hypothesis

$\emptyset$	–	Empty set
R_x	–	Rank of CRS scores
R_y	–	Rank of VRS scores
$t_{(x)}$	–	Number of independent rank
$t_{(y)}$	–	Number of dependent rank
H	–	Kruskal walli statistic
H_c	–	Kruskal walli corrected Statistics
N	–	Total number of observations
n_i	–	Number of DMUs
R_i	–	Rank sum for each treatment
p	–	length of window
w	–	number of window
k_w	–	number of periods
X_{k_w}	–	Matrix of input for window
Y_{k_w}	–	Matrix of output for window

LIST OF ABBREVIATION

AHP   –   Analytical Hierarchy Process

BCC   –   Banker Cooper Charnes

CRS   –   Constant Return to Scale

CCR   –   Cooper Charnes Rhodes

CAPEX   –   Capital Expenditure

COLS   –   Corrected Ordinary Least Square

CI   –   Consistent Index

CR   –   Consistent Ratio

CU   –   Catch Up Effect

DEA   –   Data Envelopment Analysis

DRS   –   Decreasing Return to Scale

DMU   –   Decision Making Unit

FS   –   Frontier Shift

FTE   –   Full Time Equivalent

GWH   –   Giga Watt Hour

IRS   –   Increasing Return to Scale

MCF   –   Maximum Cross Reference

MPI   –   Malmquist Productivity Index

MPSS   –   Most Productive Scale Size

MCDA   –   Multi Criteria Decision Analysis

NIRS   –   Non Increasing Return to Scale

NDRS   –   Non Decreasing Return to Scale

NWSC   –   National Water and Sewage Corporation

NRW   –   Narrow Width

OLS   –   Ordinary Least Square

PTE   –   Pure Technical Efficiency

RI	—	Random consistency index
SE	—	Scale Efficiency
SBM	—	Slack Based Model
WSBM	—	Weighted Slack Based Model
SESB	—	Sabah Electricity Sdn Berhad
SESCO	—	Sarawak Electricity Supply Corporation
SFA	—	Stochastic Frontier Analysis
TE	—	Technical Efficiency
TDC	—	Transmission Distribution Cable
TDS	—	Transmission Distribution Substation
TNB	—	Tenaga Nasional Berhad
TNE	—	Total Number of Employee
TNC	—	Total Number of Customer
TUS	—	Total Unit Sold
TRV	—	Turn Over
UTM	—	Universiti Teknologi Malaysia
VRS	—	Variable Return to Scale
OPEX	—	Operating Expenses

CHAPTER 1

INTRODUCTION

1.1   Introduction

The key requirement in using operations research models is the recognition of a "utility function" which blends all variables relevant to a decision problem into a single variable which is to be optimized [1]. Fundamentally, the concept of a utility function is the aspect that it should represent the decision-maker's perceptual experience of the relative importance of the variables involved rather than being regarded as uniform across all decision-makers or externally brought down [2]. The problem, of course, is that the resulting utility functions may bear no relationship to each other and it is therefore difficult to make comparisons from one decision context to another. Hence, not only may it not be possible to compare two different decision-makers but it may not be possible to compare the utility functions of a single decision-maker from one context to another [3].

A conventional way to combine variables in a utility function is to use a cost/effectiveness ratio, called an "efficiency" measure [4]. It measures utility by the "cost per unit produced". On the surface, that would appear to make comparison between two contexts is made possible by comparing the two cost/effectiveness ratios. The problem, though, is that two different decision-makers may place different emphases on the two variables. It also must be accepted that effectiveness will by and large mean consideration of a number of products and services and costs a number of sources of costs. Cost/effectiveness measurement requires combining the sources of

cost into a single measure of cost and the products and services into a single measure of effectiveness [5].

Again, the problem of different emphases of importance must be acknowledged. This is especially the case for the several measures of effectiveness. But it may also be the case with the several measure of costs, since some costs may be regarded as more important than others even though they may all be measured in monetary unit. When some costs cannot be measured in money terms, the problem is combined. More general, instead of costs and effectiveness, the variables may be identified as "input" and "output". The efficiency ratio is then no longer qualified as cost/effectiveness but as "output/input", but the issues identified above are the same [6].

This issue can be illustrated by evaluating hospital performance. Effectiveness here is the extent to which hospital services meet the expectations or goals set by the organization served [7]. It is measured by several services, which are the outputs of hospital operations. The outputs are Patient-days of service under Medicare, Patient-days of service not under Medicare. The inputs are numbered of nurses trained and number of interns trained. Input's measures are numbered of full-time equivalent (FTE), non-physician personnel. The amount spent on supplies, number of bed-days available, to which evident costs can be assigned, but they are also represented by measures of the populations served.

Efficiency measures the hospitals ability to transform its inputs (resources and demands) into production of outputs (services). The objective in doing so is to optimize the balance between the level of outputs and the level of inputs [8]. The success of the hospital, like that of other organizations, depends on its ability to behave both effectively and efficiently [9]. The issue at hand, then is how to combine the several measures of input and output into a single measure of efficiency [10]. The method we will examine is that called "data envelopment analysis".

1.2   Frontier benchmarking methods

Frontier Benchmarking places a greater emphasis on performance variations relative to the top performing firms, than does ordinary least square (OLS) [11]. Frontier based benchmarking methods estimate (using regression techniques) or identify (using DEA techniques) the efficient performance frontier for a sample of firms [12]. The efficient frontier is the benchmark against which the relative performance of a firms is measured [13]. Given a particular sample, all companies are assumed to be able to operate at an optimal efficiency level which is determined by the most efficient companies in the sample. These efficient companies are sometimes referred to as the "peer firms" and determine the efficiency frontier [14]. The companies that form the efficient frontier use minimum quantity of inputs to produce the same quantity of outputs. The distance to the efficiency frontier provides a measure of inefficiency [15].

The frontier based benchmarking methods identify or estimate the efficient performance frontier from best practice in a sample of DMUs. The efficient frontier is the benchmark against which the relative performance of DMUs is measured. The main frontier benchmarking methods are Data Envelopment Analysis (DEA), Corrected Ordinary Least Squares (COLS), and Stochastic Frontier Analysis (SFA) [16,17]. DEA is based on linear programming [18] while COLS and SFA are statistical techniques [19] .

Data Envelopment Analysis (DEA) is a nonparametric technique in operations research for evaluating the relative efficiency of organizational units such as universities departments, schools, hospitals, shops, bank branches, courts, countries and similar instances where there is a relatively homogeneous set of units [20, 21]. Non parametric methods do not suppose a functional form/shape for the frontiers; nevertheless they do not provide a general relationship (equation) relating output and input. These organizational units are called decision making units (DMUs). The definition of DMU is generic and flexible [22]. In economics terms DEA is employed to estimate production frontiers. In another words it is used to estimate production efficiency of DMUs [23].

There are also parametric techniques which are used for the estimation of production frontiers, see Lovell [24] for an early survey. These techniques required that the shape of the frontier be estimated in advance by specifying a particular function relating output to input [25]. One can also combine the relative strengths from each of these approaches in a hybrid method , where the frontier units are first identified by DEA and then a smooth surface is fitted to these. This allows a best-practice relationship between multiple outputs and multiple inputs to be calculated [26].

The framework has been adapted from multi-input, multi-output production functions and applied in many industries [27]. DEA develops a function whose form is determined by the most efficient producers. This method differs from the Ordinary Least Squares (OLS) statistical technique that bases comparisons relative to an average producer [28]. Like Stochastic Frontier Analysis (SFA), DEA identifies a "frontier" on which the relative performance of all utilities in the sample can be compared [29]. DEA benchmarks firms only against the best producers [30]. It can be characterized as an extreme point method that assumes that if a firm can produce a certain level of output employing specific input levels, another firm of equal scale should be capable of doing the same. The most efficient producers can form a 'composite producer', allowing the computation of an efficient solution for every level of input or output. Where there is no actual corresponding firm, 'virtual producers' are identified to make comparisons" [31].

DEA assigns weights to the inputs and outputs of a DMU that gives it the best possible efficiency. It thus arrives at a weighting of the relative importance of the input and output variables that reflects the emphasis that appears to have been placed on them for that particular DMU. At the same time, though, DEA then gives all the other DMUs the same weights and compares the resulting efficiencies with that for the DMU of focus [32]. In its simplest form where a DMU has a single input and a single output efficiency is defined simply as:

$$\text{Efficiency} = \frac{\text{Output}}{\text{Input}}$$

More typically a DMU has multiple unequal inputs and outputs and this complexity can be incorporated into an efficiency measurement by defining the efficiency as:

$$\text{Efficiency} = \frac{\text{Weighted sum of output}}{\text{Weighted sum of input}}$$

This definition requires a set of weights to be determined and this is difficult, particularly if a common set of weights to be applied across the set of DMUs is sought [33]. This problem can be resolved by arguing that each DMU can have its own particular value systems and therefore may legitimately define their own peculiar set of weights [34, 35].

1.3 Background of the research

The concept of Frontier analysis was first proposed by Farrell [36] and put the foundation of DEA. Subsequent development followed by the publication of the article entitled "Measuring the efficiency of decision making units" by Charnes, Cooper and Rhodes (CCR) [37] thirty three years ago. After the publication of CCR [37] envelopment model, a number of useful extensions have appeared in the DEA literature, see Seiford [38], Emrouznejad et al. [39], and Gattoufi et al. [40], to mention a few of them. The extensions allow analyst to easily investigate efficiency changes over multiple time periods, and to measure congestion treat both non discretionary and categorical inputs and outputs and to incorporate or ancillary managerial information. DEA and its extensions have been widely used over the past three decades.

Four basic questions need to be addressed by management of DMUs who are interested in better ways of evaluating effectiveness and efficiency of DMUs.

(i) What level of productivity or services should be obtained with available resources?

(ii) What target is required , and where are the shortcomings?

(iii) What resources acquisitions or redistributions are needed to achieve maximum improvement in efficiency and effectiveness?

(iv) How can management system by changed to improve the identification and correction of factors which limit the efficiency of DMUs?

The answers to these questions provide an opening to the issues that will be addressed in this thesis. One of the important areas of DEA that attract our attention is window analysis. This field is relatively rare in DEA literature, see Emrouznejad et al. [39], Gattoufi et al. [40] and Seiford [38]. When applying DEA, an important rule of thumb is that the number of DMUs is at least twice the sum of the number of inputs and that number plus outputs, otherwise, the model may produce numerous relatively efficient units and decrease discriminating power [31,41].

To resolve this difficulty, DEA window analysis [42] was introduced to increase the number of DMUs. This approach makes it feasible to observe how each DMU performs in different periods. In another development, Weyman and Jones [43] indicated that as the ratio of the total sum of input and output over the total number of DMU rises the ability of the model to discriminate amongst the DMUs falls significantly, since it becomes more likely that any given DMU will find some set of output and input weights which will make it appear efficient.

$$\text{ratio} = \frac{\sum_{i=1}^{m} x_{ij} + \sum_{r=1}^{s} y_{rj}}{\sum_{j=1}^{n} DMU_j}$$

DEA window analysis is useful for examining the stability and trend properties of DMUs' performances over time. It is used to detect performance trends of a DMU over time. Each DMU in a different period is treated as if it was a different DMU. In doing so, the performance of a DMU in a particular period is contrasted with its own performance in other periods in addition to the number of other DMUs. This increase

the number of data points in the analysis, which can be useful when dealing with small sample sizes.

A DEA window analysis works on the principle of moving average [44, 45]. A window analysis approach represents one area for further research in DEA. For example, the problem of choosing the width for a window (and the sensitivity of DEA solutions to window width) is currently determined by empirical approach. Similarly, the theoretical implications of representing each DMU as if it were a different DMU for each period in the window remain to be worked out in full detail [46].

At this point we can limit our research problem to the issue of selection of window width. We now propose a Multi Criteria Decision Analysis (MCDA) especially Analytical Hierarchy Process (AHP) to select the window width, for the following reasons: AHP assists decision makers to detect one decision alternative that best fits their end and their understanding of the problem. It renders a comprehensive and intellectual framework for structuring a decision problem, for representing and quantifying its elements, for relating those elements to overall goals, and for evaluating alternative solutions. AHP commutes these evaluations to numerical values that can be processed and compared over the entire range of the problem. A numerical weight or priority is deduced for each element of the hierarchy, allowing diverse and often incomparable elements to be compared to one another in a rational and consistent way. This potentiality distinguishes the AHP from other decision making techniques.

The strength of this approach is that it organizes tangible and intangible factors in a systematic way and provides a structured and yet relatively simple solution to the decision-making problems [47]. In addition, by breaking a problem down in a logical fashion from the larger to the smaller one in descending but gradual steps, one is able to, through simple paired comparison, judge the small to the large.

This research represent recent significant developments, more remain to be done however, the main factor and condition that inspire this research work is sequel to Charnes et al. [44, 45, 48]. Window analysis applications over the past decades have improved significantly to the reliability of managerial and policy implications of DEA

practice [41]. We believe that the findings of this research will enrich future practice of DEA. The selection of this problem is necessarily partial in both senses of the word. First, it can only deal with a subset of the available problems, and secondly the topics chosen for the research work represent some of the current research interests of the researchers.

1.4   Problem statement

The identified problem are stated in the following manner:

(i) What is the impact on efficiency scores for selecting a window width that is small or large figure?

(ii) What is the sensitiveness of DEA solution to the selected window width?

The aim of this research is to originate a methodology for selecting a window width in DEA window analysis.

1.5   Research objectives

In order to achieve this, it will be necessary to formulate conceptual framework in order to extend what can be accomplished by empirical application and simulation studies. Based on the problem statement, the objectives of the research are:

(i) To develop an efficient and effective AHP based method for selection of window width.

(ii) To apply the method to electrical energy modeling.

(iii) To analyze the performance of AHP.

1.6   Scope and limitation

This research is covering an area of study for the selection of window width in DEA window analysis, that is based on empirical approach. The research work is intended to originate and suggest an approach that could formalize the traditional thinking and be better than guess work. However, it is not hitch free due to an inadequacy of data. As the data requirement is very difficult. Large quantity of data on inputs and outputs are needed. Small numbers of DMU in different time periods are required. It demands relatively large sample size of panel data for each time period. The panel data has to be balanced (same DMU appeared in every period, for a sample sheet see panel data sheet on page 82), which is frequently not easy to acquire.

1.7   Significance of findings

This research work has attempted to open a fresh direction in the realizing and rating of DEA window analysis technique through two complementary approaches. The first approach has been to attempt a conceptual clarity by means of a newly conceptual framework for the key concepts. The second approach has been the development of a computationally tractable, mathematical decision making technique which measures pragmatic performance data to test the conceptual distinctions established in the newly framework. These two approaches are represented in the four contributions extended in this thesis.

The first contributions of this research work is the explication and introduction of a methodology for the selection of window width in DEA window analysis. This approach is an application of Analytic Hierarchy Process (AHP), which helps decision makers to choose one best decision alternative among finite number of decision alternatives that best accommodates their goal and understanding of the problem, it is a process of organizing decisions that people are already dealing with, but trying to do in their heads. It provides a comprehensive and intellectual framework for structuring a decision problem, for representing and quantifying its elements, for relating those

elements to overall goals, and for evaluating alternative solutions. The method has three major strengths.

- It helps decision makers find one decision alternative that best suits their goal and their understanding of the problem. It provides a comprehensive and rational framework for structuring a decision problem, for representing and quantifying its elements, for relating those elements to overall goals, and for evaluating alternative solutions.

- It converts these evaluations to numerical values that can be processed and compared over the entire range of the problem. A numerical weight or priority is derived for each element of the hierarchy, allowing diverse and often incommensurable elements to be compared to one another in a rational and consistent way. This capability distinguishes the AHP from other decision making techniques.

- In the final step of the process, numerical priorities are calculated for each of the decision alternatives. These numbers represent the alternatives' relative ability to achieve the decision goal, so they allow a straightforward consideration of the various courses of action.

The second contribution of this research work is the creating by mental acts of DEA window analysis input oriented multiplier model under CRS assumptions see Equation (3.6). On the other hand, Asmild et al. [48] has shown the DEA window analysis input oriented envelopment model under CRS assumptions. This is one of the major contributions of this research work. The formulation is called the dual in linear programming terminology.

The third contribution of this thesis is the introduction of the Kruskal-Wallis test statistics for population sample. This is called non homogeneous environment. TNB is big of operation when compared to SESB and SESCO. The environment where TNB operates is that its turnover is an extraordinary amount. This gave over SESB and

SESCO. Whenever appraisal is carried out between these three DMUs the efficiency assessment incline to be bias over TNB, because of non homogeneous environment. The efficiency assessment inclines to be overrated or under rated. Therefore, if we subjected all DMUs under test of significance, the result will confirm the status of sample if they are from the same population. This is one of the pitfalls of DEA [49].

The fourth and final contribution of this thesis is the application of this framework to the evaluation of Malaysian electricity companies. This demonstration of the methodology provides a concrete evidences of unambiguous conclusion that conceptual model for the selection of window width is a superior approach when compared with empirical approach. The benefit of this finding cannot be over emphasized. It has opened up a new era in DEA window analysis technology for further research in the field of DEA window analysis. The result has unveiled a procedure that replaces a guess work in the selection of window width. The full significance of this conclusion which we are unaware now would be discovered in a mere future.

1.8   Organization of the thesis

The thesis is prepared into six chapters. In the first chapter is the introduction that traces the development of the research problem. That is a selection of window width by an empirical approach. Furthermore, the reason why AHP is an attractive methodology for solving the identified research problem is stated. The basic concepts of the main methodology with brief history of its origins are given to support it uses as the main methodology in the thesis.

In the second chapter, we review the current state literatures' empirical approach in the selection of window width. The literature also elaborates on some DEA extension's models, such as the slack based model, weighted slack based, radial model; It weighted non radial model, super efficiency model, cost efficiency model, window analysis and Malmquist productivity index are presented to support theoretical

model development. The chapter also reviewed parametric model such as a stochastic frontier model, ordinary least square and corrected least square method. The chapter concludes with definition of terms.

In the third chapter of this thesis, we developed an efficient and effective methodology for selection of window width. The research problem is then articulated and figured out. A numerical example of the model is also demonstrated, in order to establish concrete evidence. A newly DEA window analysis output oriented envelopment multiplier model was formulated and presented. A window width was selected and applied to electrical energy modeling.

In the fourth chapter of the thesis is the presentation of the data validation and modeling processes of electrical energy. A set of Panel data is acquired, and descriptive statistics are summarized. The relationship between inputs and output were shown using the spearman correlation coefficient. The set of DMU were confirmed to belong to the same population using Kruskal-Wallis test. These hypotheses were shown to be beyond doubt on these data to authenticate its genuineness.

In the fifth chapter of the thesis, we show the performance analysis and comparison of AHP based model with an empirical approach. The result is presented in two ways. Firstly, we analyze the results bring forth by the application of our formulated framework, and secondly. We examine the results produce by an empirical approach. In each case, a sensitivity analysis is conducted upon which a conclusion is made.

The last chapter comprises the summary and conclusion of the whole thesis. The analyses of the two approaches are summed up in this chapter. We try to compare the areas of common similarity between the two methods, with tables and observations. It is visible to see the shortcoming of guess work from these tables. The chapter enumerates three solid contribution to knowledge in the field of DEA window analysis. The contributions are highlighted one after the other. Future extension is possible, to examine whether frontier has shifted and catch up effect taken place. In doing so, one should be able to figure out whether technology changes have taken place within the period of a case study.

1.9   Assumptions

DEA makes a range of homogeneity assumptions about the DMUs under evaluation. Overall the DMUs are believed to be similar in a number of ways:

  (i) All DMUs viewed are homogeneous, i.e., they all have the same types of inputs and outputs, and are independent, i.e., no constraint attaches input and output levels of a DMU with the inputs and outputs of other DMUs. In other words all DMUs are presumed to be undertaking similar activities and making comparable products or services so that a common set of outputs can be specified [50].

 (ii) All DMUs are supposed to be using a mutual technology in their operations. The technology in practice is having the same connection with two or more DMUs [51].

(iii) Accessibility of similar range of resources to all DMUs. These includes: Staff, raw materials, work-in-progress, equipments and machinery, although, if for instance, different equipment is being hired, which can be priced then comparisons can still be made providing the resources can still be brought to a common denominator such as cost.

(iv) All DMUs carry out operations in a similar environment. This is because the external environment generally impacts upon the overall performance of a unit. However, this assumption can hardly be met, and as such environmental variables are often brought into the analysis to supplement the input/output set.

1.10   Summary

In the chapter, the thesis is introduced. It direct the attention of the subject matter and basis for the work done in subsequent chapters. A few of the topics discussed here are at the introductory level and will be explained in details. This succeeding chapter reviewed the literature of the thesis.

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CATALYTIC OXIDATIVE CHEMICAL TREATMENT FOR THE REMOVAL OF
ELEMENTAL MERCURY ON CARBON STEEL (SAE J429) SURFACE

FARAH ILYANA
KHAIRUDDIN

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Universiti Teknologi Malaysia

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Specially for my beloved family and friends

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ABSTRACT

In this study, mercury contaminated carbon steels was prepared using droplet and physisorption methods. Various oxidants were applied to oxidize the mercury element and the oxidized mercury and the iron leaching were analyzed using Atomic Absorption Spectrometer (AAS) for data collections. The effect of oxidant system of KI/I_2, peracetic acid, different conditions of experiment namely heating, stirring, left at room temperature, the presence of catalysts and the addition of imidazoline based corrosion inhibitor were investigated. The experiment revealed the oxidant system of $1H_2O_2:1CH_3COOH$ (peracetic acid) ratio as the best to remove 96.43% physisorbed Hg and 96% droplet Hg from carbon steel surfaces under ambient temperature and soaking for 5 hours. The total iron leached detected under the optimum condition from used carbon steel contaminated with physisorp Hg and droplet Hg were 21.45 ppm and 22.98 ppm respectively. Interestingly, the presence of Ru/Mn (25:75)/Al_2O_3 catalyst calcined at 1000°C with peracetic acid as oxidant could further remove 99% of Hg for CS-physisorbed-Hg and 98.71% for CS-droplet-Hg resulting in 19.71 ppm and 19.62 ppm respectively iron leached in 3 hours. FESEM illustrated the catalyst surface is covered with small and dispersed particles with undefined shape. From FESEM-EDX analysis, Mn species were detected in all the catalysts tested. The X-Ray Diffraction (XRD) analysis revealed that the catalyst is crystalline and Mn species is believed to be the active species for the catalysts. Nitrogen Gas Adsorption (NA) analysis showed that both fresh and spent catalysts are of mesoporous material with Type IV isotherm and type H_3 hysteresis loop.

ABSTRAK

Dalam kajian ini, keluli karbon tercemar merkuri telah disediakan menggunakan teknik titisan dan fizijerapan. Berbagai bahan pengoksida diaplikasikan untuk mengoksida elemen merkuri dengan menggunakan sistem pengoksidaan KI/I_2 dan asid perasetik. Kondisi eksperimen yang berbeza iaitu pemanasan, pengacauan, dibiarkan pada suhu bilik, dengan kehadiran pemangkin dan penambahan perencat kakisan berasaskan imidazolin juga dikaji. Merkuri yang teroksida dan ferum terlarut telah dianalisa menggunakan Spektroskopi Serapan Atom (AAS) untuk pengumpulan data. Eksperimen membuktikan bahawa sistem pengoksidaan $1H_2O_2:1CH_3COOH$ (asid perasetik) adalah yang terbaik untuk menyingkirkan 96.43% Hg-fizijerapan dan 96% Hg-titisan daripada permukaan karbon keluli pada suhu bilik dan direndam selama 5 jam. Ferum terlarut bagi Hg-fizijerapan adalah 21.45 ppm dan 22.98 ppm bagi Hg-titisan. Menariknya, kehadiran mangkin Ru/Mn $(25:75)/Al_2O_3$ yang telah dikalsinkan pada suhu 1000°C dengan asid perasetik sebagai bahan pengoksida boleh menyingkirkan 99% Hg bagi Hg-fizijerapan manakala bagi Hg-titisan adalah 98.71% dengan ferum terlarut sebanyak 19.71 ppm dan 19.62 ppm selama 3 jam. Mikroskop Pengimbas Elektron Emisi Medan (FESEM) menunjukkan permukaan pemangkin diselaputi dengan zarah-zarah halus yang mempunyai bentuk yang pelbagai. Daripada analisis Spektroskopi Sinar-X Penyebar Tenaga (EDX) spesis Mn telah dikesan bagi semua mangkin yang telah diuji. Analisis Pembelauan Sinar-X (XRD) pula menunjukkan mangkin adalah dalam bentuk kristal dan spesis Mn adalah spesis aktif bagi mangkin-mangkin tersebut. Penyerapan Nitrogen (NA) menunjukkan mangkin yang baru dan yang telah digunakan masing-masing mempunyai ciri bahan mesoporous dan *Isotherm Jenis IV* juga histerisis lengkokkan H_3.

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LIST OF ABBREVIATIONS

AAS	-	Atomic Absorption Spectroscopy
BET	-	Brunauer–Emmett–Teller
CI	-	Corrosion inhibitor
CS-droplet-Hg	-	Carbon steel droplet Hg
CS-physisorbed-Hg	-	Carbon steel physisorbed Hg
DOE	-	Department of Environment
DOC	-	Dissolved organic carbon
di-PAA	-	Diperacetic acid
EDX	-	Energy dispersive X-ray spectroscopy
FESEM microscopy	-	Field emission scanning electron
LME	-	Liquid metal embrittlement
MHS-AAS Absorption	-	Mercury-Hydride System, Atomic Spectroscopy
PAA	-	Peracetic acid
SAMMS	-	Self-assembled mercaptan groups on mesoporous silica
SS	-	Sewage sludge
TBHP	-	Tert-butyl hydroperoxide
XPS	-	X-Ray photoelectron spectroscopy
XRD	-	X-ray Diffraction

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CHAPTER 1

INTRODUCTION

1.1   History of Mercury

Mercury concentrations in natural gas can range from below 1 ng m^{-3} to greater than 1000 μg m^{-3} depending on the location, the well or the process and is measured using amalgamation atomic fluorescence spectrometry. Mercury is of great concern receiving a major focus due to its unique high toxicity, volatility, and persistence in the environment and easiness of bioaccumulation. Organic forms of mercury are more toxic than inorganic forms, but it is possible for inorganic mercury to be biologically methylated. Methyl mercury has high affinities for fatty tissues in organisms and can accumulate through food chain to higher toxic levels within those organisms. Therefore, it is important to have a strict control on inorganic mercury leaching from mercury-containing wastes (Jian *et al*., 2002).

Elemental mercury (Hg°), although is a metal, at normal temperatures, it is in liquid form. Thus, because of this unique property, plus its high specific gravity and electrical conductivity, has brought about its various types of laboratory equipment and instruments extensive use in the industries. The elemental mercury is also extremely dense which is 13.5 times denser than liquid water under ambient conditions. This high density, low saturation vapor and high surface tension control

the immediate behavior of the releasing of elemental mercury on land surface (Turner, 1992).

Mercury can exist in three oxidation states, which is Hg° (metallic), Hg_2^{2+} (mercurous) and Hg^{2+} (mercuric). These oxidation states will determine the properties and behavior of the mercury. Mercury (Hg), is one of the most toxic heavy metals commonly found in the global environment including lithosphere, hydrosphere, atmosphere and biosphere. Cycle of three-oxidation states of Hg to the environment is allowed by a series of complex chemical transformations allows. Most of the Hg encountered in all environmental media (water/soil/sediments/biota) is in the form of inorganic mercuric salts and organomercurics, with the sole exception of atmosphere. The mercuric salts $HgCl_2$, $Hg(OH)_2$ and HgS are the prevalent forms existing in the environment and CH_3HgCl and CH_3HgOH are main forms of organic compounds, together with other organomercurics (eg: dimethylmercury and phenylmercury) existing in small fractions (USEPA, 1997)

1.2 Mercury Flow through Petroleum and its Scenario to Environment

The mercury from industries and power plants is emitted primarily as mercury vapour. This vapor consists mainly of elemental mercury and dimethyl mercury. It is difficult to say which volatile compound dominates the discharge process. Mercury species other than elemental Hg and $(CH_3)_2Hg$ can also contribute. Most mercury is emitted as dimethyl mercury with a relatively fast degradation to elemental mercury taking place in the air. Hg (O) is mobilized to the atmosphere where it is subjected to atmospheric oxidation processes to yield water soluble forms, subsequently scavenged by wet or dry deposition (Elisabeth *et al*., 2000)..

Petroleum products carry mercury from a geological reservoir and distribute mercury to the environment along their passage. This section describes the flow and trend of mercury as carried by petroleum products. More work with the more

sensitive analytical methods developed in the past few years should be performed to confirm these numbers.

Crude petroleum is identified to contain small but measurable amounts of mercury. About 16 to 18 million barrels (672 to 756 million gallons) of crude oil are consumed daily in the United States. At an average concentration of 0.41 ppm mercury and an average density for crude oil of 6.9 lbs per gallon, the lowest total amount of mercury vaporized daily is therefore 1,901 lbs. This value represents an annual discharge of 347 tons of mercury nationwide, assuming that all of the oil is combusted. As very large volumes of oil consumed, even a small concentration of mercury clearly represents a major source of atmospheric deposition of mercury.

Some natural gas regulators made before 1961 contained Hg°, which was sometimes spilled when the regulators were removed. After a large Hg° spill, the hazard can persist for a long time. In the case of natural gas regulator spills, monitoring found elevated airborne Hg^0 > 10 years after it was spilled. Spilled Hg^0 forms small beads, which spread, making a thorough cleanup difficult.

1.3   Techniques of mercury removal

Chemical leaching where the chemical separation is based upon the reactivity of mercury and employs solution leaching of the mercury-contaminated materials can do removal of mercury from metal surfaces. Solution leaching may be used to remove both elemental and inorganic forms of mercury. Most common used leaching solutions are the oxidizing acids such as nitric acid, hypochlorous acid and sulfuric acid. These oxidizing acids are used because of their ability to readily dissolve elemental and inorganic mercury (Foust, 1993). Preferred oxidizing agents are those, which are characterized as being mild, and which do not react with any of the solid material to form oxidation products, which complicate separation contamination of the solid material. In this case, iodine is a most preferred oxidant.

Removal of mercury from solid waste can be conducted by using a lixiviant consisting an aqueous solution of potassium iodide/iodine (KI/I_2) (Ebadian, 2011a). Mercury in contaminated solid wastes in the form of oxides, sulfides, elemental, and adsorbed phases is mobilized by the KI/I_2 lixiviant through oxidation and complex-forming reactions. Iodine, which is an oxidizing agent, is capable to oxidize various species of mercury including elemental mercury to mercuric iodide. While potassium iodide is a complexing agent, thus it can react with mercuric iodide to form a water-soluble compound, which has the formula of K_2HgI_4.

In addition, in order to increase mercury solubility for absorption, oxidizers such as sodium hypochlorite and hypochlorous acid have been applied to transform insoluble Hg^o to very soluble Hg^{2+} which can then be easily moved through aqueous scrubbing (Zhao *et. al*, 2008a). Elemental mercury absorption in hypochlorous acid was found to be much more reactive than hypochlorite but the mercury removal reactivity of hypochlorite increased in the presence of sodium or potassium chloride and potassium hypochlorite was found to be more reactive than sodium hypochlorite (Zhao *et. al*, 2008b and Lynn *et. al*, 1999). NaOCl strongly absorbs elemental Hg vapor even at high pH. At low pH, high concentrations of chlorine⁻ and high temperature favor mercury absorption.

One of the most established approches on removing mercury from wastewater is precipitation and coagulation/co-precipitation technology (Ebadian, 2001b). Sulfide is added to the waste stream to convert the soluble mercury to the relatively insoluble mercury sulfide form:

$$Hg^{2+}{}_{(aq)} + S^{2-}{}_{(aq)} \longrightarrow HgS_{(s)} \qquad (1.0)$$

The process usually combined with pH adjustment and flocculation, followed by solid separation. The sulfide precipitant is added to the wastewater in a stirred reaction vessel, where the soluble mercury is precipitated as mercury sulfide. The precipitated solids can be removed by gravity settling in a clarifier. Sulfide precipitation can achieve 99% removal for initial mercury levels excees of 10 mg/L

(Patterson, 1985). Approximately 10 to 100 µg/L are the lowest achievable effluent mercury concentration that appeared for various initial concentrations even with polishing treatment such as filtration. Sulfide precipitation appears to be the common practice for mercury control in many chlor-alkali plants. A 95 to 99.9% of removal efficiencies were reported well-designed and managed mercury treatment systems (Perry, 1974).

Numerous studies have been conducted on the mercury removal from aqueous medium but the most preferable technique is to use photocatalyst. Photocatalytic processes use electron-hole pairs photogenerated in semiconductors to promote redox reactions. The photocatalytic treatment for mercury (II) produces metallic mercury that deposits on the photocatalysts (Aguado *et. al*, 1995).

1.4   Problem Statement

Crude oil and unprocessed gas condensate can contain significant amount of mercury. Elemental mercury Hg^0 is independently quantified as volatile species evaporated from a single crude oil using selective trapping. Steel sorbs mercury in considerable quantity. Hg^0 both adsorbs and chemisorps to metal surfaces.

Mercury is common and naturally occurring component of petroleum. Petroleum processing often is accompanied by generation waste streams contain some mercury. These waste streams become problematic when the mercury concentration in process feeds exceeds a few ppb because of the highly toxic nature of mercury.

In gas processing, mercury damages equipment and fouls cryogenic exchangers. Pipelines that carry fluids that contain mercury can become contaminated over time and thus require special attention. The interactions of mercury with pipe surfaces affect worker health and safety strategies and impacts

operational procedures. Therefore, the wastes that contain mercury must be disposed in safe manner so that the world will not be a dangerous place to live for another generations (Wilhelm, 1999).

There are few solution used in industry to solve mercury metal presence on material surface, mostly by using inorganic acid, but it reacts with the metal surface and became corrode. Recently, technologies claimed lixiviant chemical is potential to remove mercury from metal surfaces, but it reacts with the material for example, carbon steel. The critical successfulness of the technique should be no or acceptable reaction towards the material surface, instead reacts with Hg metal. Thus, this research is proposed to suggest the most effective way to treat mercury on metal surfaces so that it can be used in the industry.

1.5   Significance of Study

In this research, peracetic acid with the addition of a potential catalyst can be used to enhance the removal of elemental Hg presence on the metal surfaces.

The removal technique via this oxidant and catalyst can remove elemental mercury that is hazardous to the environment. This will help to prevent mercury, which has been known to be causing serious impact on human health, animals, plants and also the environment. Mercury was found to produce several impacts on gas processing production. These includes, it forms amalgams with several metals, particularly carbon steel, which leads to LME. This is prevelant in pipeline welds, cryogenic components, heat exchangers and hydrogenation catalysts. Besides, it may be necessary to avoid the corrosion and clogging to the delivery pipeline. This cleaning method will certainly improve the quality and quantity of Malaysian oil manufacturing company. The utmost important, the oxidant and potential catalyst will contribute to the growth of the national economy and create green and sustainable environment. This proposed technique enables to conduct treatment of elemental mercury in the internal pipeline system.

The oxidant and the catalyst are easily prepared and environmental friendly. All the ingredients in the fabrication of both oxidant and catalyst are easily available, cheap and stable. It requires minimum modification to the already existing system and offers cost effective operating system.

1.6   Objective of Study

The objectives of this research are:

1. To develop the oxidizing agent, potential for the treatment of mercury metal presence on metal surface

2. To test the catalytic activity of the prepared catalyst for elemental mercury removal from metal surfaces

3. To optimize the catalytic oxidative reaction for elemental mercury removal

3. To characterize the prepared catalysts utilizing various analytical techniques

1.7   Scope of Research

The removal of mercury from metal surfaces will be done using five different types of oxidizing agents, which are iodine/iodide lixiviant (KI/I_2), sodium hypochlorite (NaOCl), diperacetic acid (di-PAA), peracetic acid (PAA), and tert-butylhydroperoxide (TBHP). Next, a series of alumina-supported catalyst based on ruthenium oxide doped with noble metal were prepared using wetness impregnation techniques. Meanwhile, adding the prepared catalysts to the oxidants carried out catalytic testing. Carbon steel physisorbed Hg (CS-physisorbed-Hg) and Carbon steel droplet Hg(CS-droplet-Hg) will be used in this experiment. The batch experiments will be carried out in a 100 ml glass beaker. Then, the samples that contain mercury will be analyzed using Mercury-Hydride System, Atomic Absorption Spectroscopy

(MHS-AAS) as the quantitative analytical method to determine the level of mercury after the treatment of the samples. Lastly, characterization of the catalysts will be carried out by various techniques including X-Ray Diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM) and Energy Dispersive X-Ray Analysis (EDX).

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