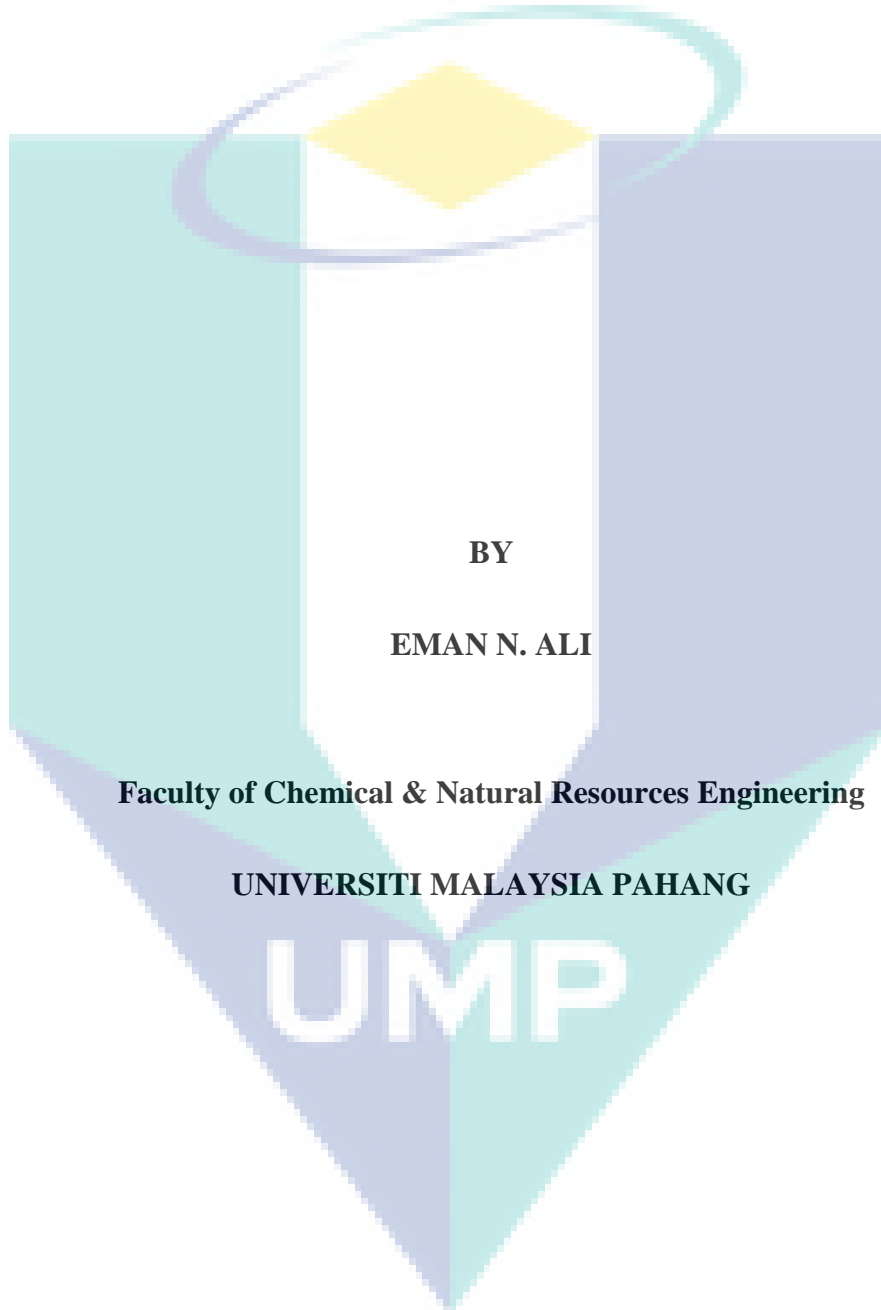


WASTE WATER TREATMENT WITH *MORINGA OLEIFERA* PRESS CAKE



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UMP

2017

ABSTRACT

WASTE WATER TREATMENT WITH *MORINGA OLEIFERA* PRESS CAKE

Key words: water treatment, *Moringa oleifera*, heavy metals.

The consumption of water nowadays is increasing but the production of wastewater from variety of industry and domestic are increasing. If this wastewater is being untreated, it will be wasted and the chemicals inside the water especially heavy metal are harmful to the living organism. There are few conventional method used to treat the wastewater but most of them are costly and hence bio-absorption with the natural coagulant is introduced. One of the multipurpose tree *Moringa oleifera*, (MO) which is environmental friendly and low cost become a choice for removal of heavy metal from wastewater. The *Moringa oleifera* press cake (MOPC) are able to remove the heavy metal but the removal efficiency for different metals need to be found out. Therefore, experiment on heavy metal removal by *Moringa oleifera* press cake was conducted. The wastewater samples and MOPC were collected from Balok River, Kuantan and Mitomasa Sdn Bhd, Kuala Lumpur respectively. MOPC was immersed in distilled water to remove the oil and different concentration of MOPC solution were made. Jar test containing six beakers with 500 mL of sample and different concentration of MOPC solution was operated at 200 rpm for 4 minutes and 40 rpm for 30 minutes. The initial and final heavy metal (Fe, Cu, and Cr) concentration were measured by Atomic Absorption Spectrometer and the removal percentage was calculated. The MOPC removed 69.99% Fe, 88.86 % Cu and 93.73% Cr at optimum concentration of 10000 ppm, 5000 ppm and 15000 ppm respectively. The MOPC also reduced 34.94% of COD and 81.6 % of turbidity. However, BOD of wastewater increase from 9.0 ppm to 18.3 ppm. Besides, pH values did not shown significant changes with the addition of MOPC. The removal of heavy metal was increased proportional with MOPC concentration until optimum removal was reached.

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ABSTRAK

Pada masa kini, penggunaan air semakin meningkat tetapi pengeluaran air sisa dari pelbagai industri dan domestik juga semakin meningkat. Jika air sisa ini tidak dirawat, ia akan dibazirkan dan berbahaya kepada organisma terutamanya bahan kimia di dalam air seperti logam berat. Sebenarnya, terdapat beberapa kaedah konvensional yang sedang digunakan untuk merawat air sisa tetapi kebanyakan kaedah ini adalah mahal dan oleh itu bio-penyerapan dengan koagulan semula jadi diperkenalkan. Salah satu bio-penyerapan adalah pokok Moringa oleifera, (MO) yang kosnya rendah dan mesra alam sekitar menjadi pilihan untuk penyingkiran logam berat daripada air sisa. Moringa oleifera biji mampat (MOPC) dapat menyerap logam berat tetapi kecekapan penyerapan bagi logam yang berbeza perlu diketahui. Oleh itu, eksperimen mengenai kecekapan bagi penyerapan pelbagai logam berat oleh MOPC telah dijalankan. Sampel air sisa dan MOPC dikumpulkan dari Balok River Gebeng, Kuantan dan Mitomasa Sdn Bhd, Kuala Lumpur masing-masing. MOPC direndam dalam air suling untuk mengeluarkan minyak dan kepekatan MOPC yang berbeza telah dicampurkan dengan enam bikar memiliki 500 mL sampel dan ujian Jar dioperasikan pada 200 rpm selama 4 minit dan 40 rpm selama 30 minit. Jumlah logam berat (Fe, Cu, dan Cr) di dalam setiap sampel diukur oleh Penyerapan Atom Spektrometer dan peratusan penyerapan telah dikira. The MOPC telah berjaya mengeluarkan 69.99% Fe, 88,86% Cu dan 93,73% Cr pada MO kepekatan optimum 10000 ppm, 5000 ppm dan 15.000 ppm.masing-masing The MOPC juga mengurangkan 34.94% daripada COD dan 81.6% daripada kekeruhan. Walau bagaimanapun, BOD air sisa menunjukkan kenaikan dari 9.0 ppm ke 18.3 ppm. Selain itu, nilai pH tidak menunjukkan perubahan yang ketara dengan penambahan MOPC. Penyingkiran logam berat meningkat berkadar dengan kepekatan MOPC sehingga penyingkiran optimum dicapai.

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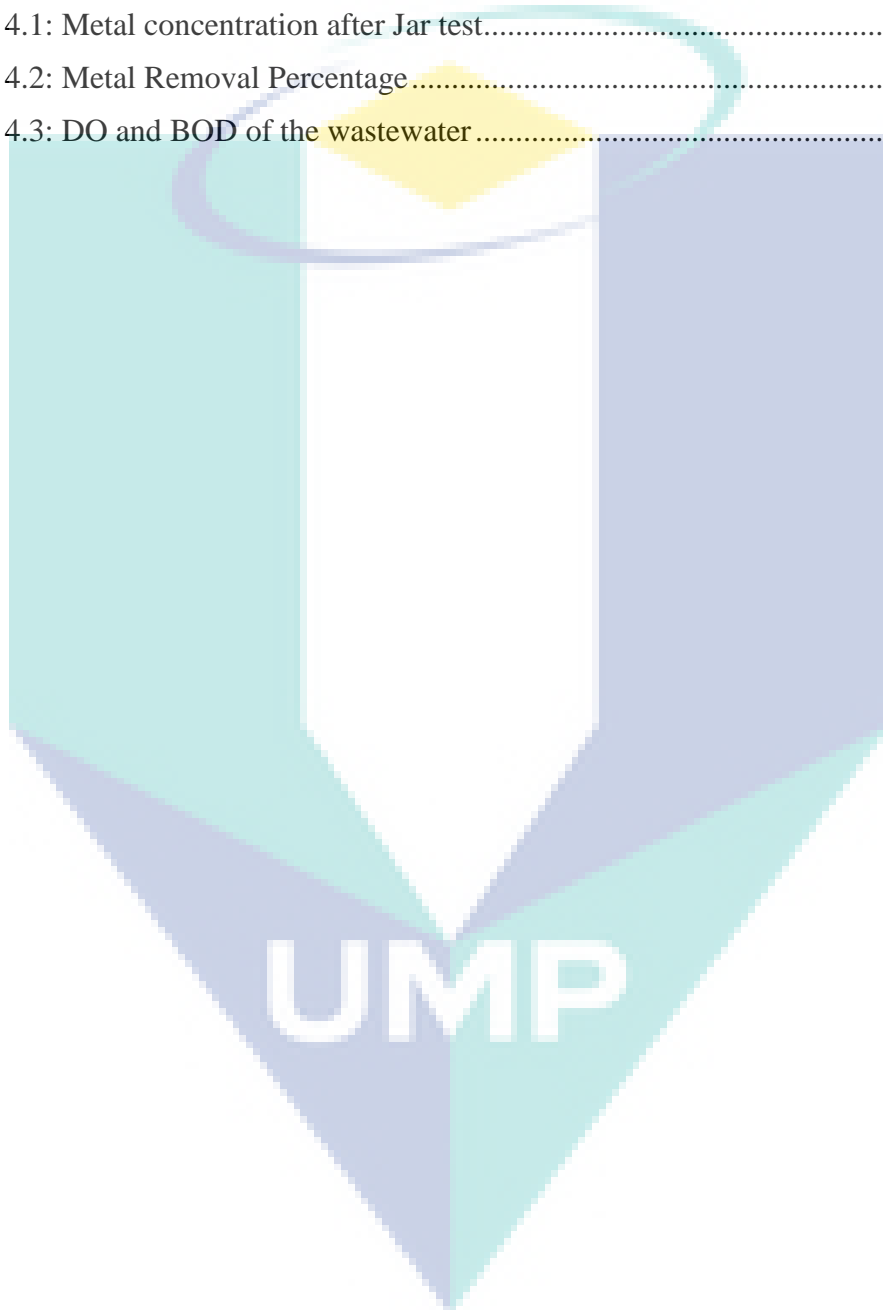
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LIST OF ABBREVIATIONS

- AAS – Atomic Absorption Spectrometry
- BOD - Biochemical Oxygen Demand
- BOD₅ - Biochemical Oxygen Demand in 5 days, mg/L
- COD - Chemical Oxygen Demand
- CMOS – Charcoal *Moringa oleifera* Seed
- DO – Dissolved Oxygen
- MC – Moisture Content
- MO – *Moringa oleifera*
- MOPC – *Moringa oleifera* Press cake
- MOS – *Moringa oleifera* Seed
- RP – Metal Remove Percentage, %
- RPC – Ram Press Cake
- WSK - Whole Seed Kernel

The logo for UMPU is a large, downward-pointing arrow shape. It is composed of four triangular sections meeting at a central point. The top-left and bottom-right sections are light blue, the top-right and bottom-left sections are a slightly darker blue, and the central point is white. The letters 'UMPU' are written in a bold, white, sans-serif font across the bottom of the arrow.

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CHAPTER 1

INTRODUCTION

1.1 Motivation and Problem statement

Water is very important for all the living organism and essential for human to survive. Recently, the consumption of water have increased due to the increase of population in the world wide. Meanwhile, large amount of wastewater containing contaminant such as suspended solid, pathogens, nutrients and heavy metals which may be harmful and dangerous are generated from the field of agriculture, domestic and industrial. If they are kept untreated, safely disposed back to the sea or reused of the wastewater will be prevented (Paula et al., 2014). From World Health Organisation 2004, drinking water supplies around the world have been identified to have thousands of chemicals considered potentially hazardous to human health at relatively high concentrations (Vikashni et al., 2012). Besides, pollution of water has received special attention as an environmental issue and cause shortage of water supply especially in the developing countries and third world countries which have inadequate financial resources (Ali et al., 2009).

One of the contaminant in wastewater is the heavy metal. These wastewater with heavy metals are mainly contributed by anthropogenic sources like electronics assembly and fabrication, battery manufacturing, paper and pulp industries, metal smelters, textiles, usage of fertilizers and pesticides, and mining activities (Kalavathy and Miranda, 2010). There are mainly 59 elements of heavy metal where cadmium (Cd), chromium (Cr), copper (Cu), lead (Pb) and zinc (Zn) are considered highly toxic (Vikashni et al., 2012). Even at low concentrations, these heavy metals can be toxic to organisms, including humans (Obuseng et al., 2012). For instance, Cadmium may cause itai-itai disease, high blood pressure and kidney damage while copper

may cause Wilson disease (Vikashni et al., 2012). Cardiovascular effects, gastrointestinal effects, and hematological effects might be happened for the excess ingest of the chromium (Agency for the Toxic Substances & Disease Registry, 2011). Overload of iron also believed to cause conjunctivitis, choroiditis and retinas particularly (Sajidu et al., 2005). To avoid these diseases, it is important to treat the wastewater to remove heavy metal.

Conventional methods used to remove dissolved heavy metal ions from wastewaters include ultra-filtration, reverse osmosis, ion exchange, solvent extraction, sedimentation and chemical precipitation (Obuseng et al., 2012). However, most of these methods has some disadvantages such as incomplete metal removal and toxic sludge disposal or treatment problem (Kalavathy and Miranda, 2010). For instance, excess concentration of aluminium sulphate (alum) which as a chemical coagulant in water treatment has been reported to cause Alzheimer's or other neuro-degenerative diseases (Paula et al., 2014). Cost of the chemical used in conventional method are too expensive for most of the developing country. Besides, country like Malaysia which having frequently land development and rainy season may cause increasing in the cost of water treatment due to unstable quality of water and high turbidity (Ali et al., 2009).

To solve this problem, natural coagulants which are more economical and environmental friendly are being researched. Natural coagulants are biodegradable coagulants and can be locally grown. Moreover, they produce less sludge and are safer for human compare to chemical coagulants. Three example of the natural coagulants are being tested by Kazi and Virupaksi (2013) which are *Cicer arietinum*, *Seed Moringa oleifera* and *Cactus Opuntiaficus* (Paula et al., 2014). It was found that both the removal of turbidity by *Cicer arietinum* and *Seed Moringa oleifera* are 90% and 82.02% respectively (Paula et al., 2014). From the research, both leave and latex of *Calotropisprocera* can be used in bioremediation of heavy metals (Patil et al., 2013). Sago (*Metroxylon* spp) is found to be one type of coagulant to remove turbidity and heavy metal which grown in Malaysia (Aziz et al., 2000).

Since *Moringa oleifera* is one of the best natural coagulant discovered yet, it can be used to replace the conventional methods widely all around the world. It is a small tree which is fast growing and can be reach to 12 meters in height (Araújo et al.,

2013). *Moringa oleifera* is one type of tropical multipurpose tree which is adaptable to semi-arid climates. Leaves and seeds of *Moringa oleifera* are widely used in water treatment which have no significant side effect as its' non-toxic and biodegradable characteristics (Ali et al., 2014). From the research, MO show the coagulation activity which are similar to the alum (Bodlund, 2013). The MO contain cationic polyelectrolytes which have proved to be effective in the treatment of water, as a substitute for aluminum sulfate (Araújo et al., 2013). The flocculation activities of MO to adsorb the metal cations are based on the electrostatic patch charge mechanism (Muyibi et al. 2002). The use of *Moringa oleifera* in water treatment also do not have significant effect on pH or increasing BOD (Sajidu et al., 2005).

Hence, the main problem is the increase in consumption of water meanwhile high amount of wastewater are generated from variety of industries. These wastewater contain chemical such as heavy metal which may cause variety of disease and need to be treated so that the water before release to sea or reuse to avoid environmental issue. The conventional method for the treatment are high cost and may containing toxic sludge which is hard to manage. Theoretically, *Moringa oleifera* press cake is one of the method for removal of heavy metal from wastewater. However, the efficiency of *Moringa oleifera* for removal of different type of heavy metal need to be determined.

1.2 Objectives

The following are the objectives of this research:

- To determine the removal of iron (Fe), copper (Cu) and Chromium (Cr) from wastewater by using *Moringa oleifera* press cake.
- To determine the removal efficiency of heavy metal by the *Moringa oleifera* press cake.

1.3 Scopes of study

The following are the scope of the research:

- Construction of experiment for removal of heavy metal (Fe, Cr, Cu) with *Moringa oleifera*.
- Experimental analysis of performance and efficiency of *Moringa oleifera* press cake to remove heavy metal from wastewater by using atomic absorption spectrometry (AAS).
- Experimental analysis of performance and effect of *Moringa oleifera* press cake on pH, turbidity, COD and BOD of wastewater by using pH meter, turbidity meter, spectrophotometer, HACH DR/2400 @ 2800 and dissolved oxygen meter respectively.

1.4 Main contribution of this work

Moringa oleifera can be applied in the treatment of the water. Owing to this application, this research contributes to the treatment of wastewater by removing heavy metal and reducing turbidity by using the *Moringa oleifera*. This research could determine the ability of *Moringa oleifera* Press Cake in removing the heavy metal concentration of the wastewater. The optimum dosage and concentration of MOPC used can be determined in this research.

1.5 Organization of this research

The structure of the remainder of the thesis is outlined as follow:

Chapter 2 provides a description and applications of the *Moringa oleifera*. Besides, it also described and analysed the research done by the preview researcher in wastewater treatment by using *Moringa oleifera*. In addition, other type of the natural coagulant which have the heavy metal removal ability are discussed and compared.

Chapter 3 gives a review of the pre-treatment of the *Moringa oleifera* press cake. Then, the detail on the Jar test are explained. Besides, review of using atomic absorption spectrometry (AAS), pH, turbidity, COD and BOD of wastewater by using pH meter, turbidity meter, spectrophotometer, HACH DR/2400 @ 2800 and dissolved oxygen meter are shown. Finally, the equation used in the calculation are shown.

Chapter 4 is devoted to the result of the heavy metal removal percentage of the experiment. The findings were tabulated and related graph were plotted. Discussion of the result were done to obtain the related information and problem faced so that recommendation can be made for future study.

Chapter 5 draws together a summary of the thesis and suggestion to improve the work in the future.



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CHAPTER 2

LITERATURE REVIEW

2.1 *Moringa oleifera*

Moringa oleifera (Figure 2.1) is tropical tree from Moringaceae family with multipurpose uses and grow mostly at dry and hot climate such as India (Pandey et al., 2011; Bodlund, 2013).

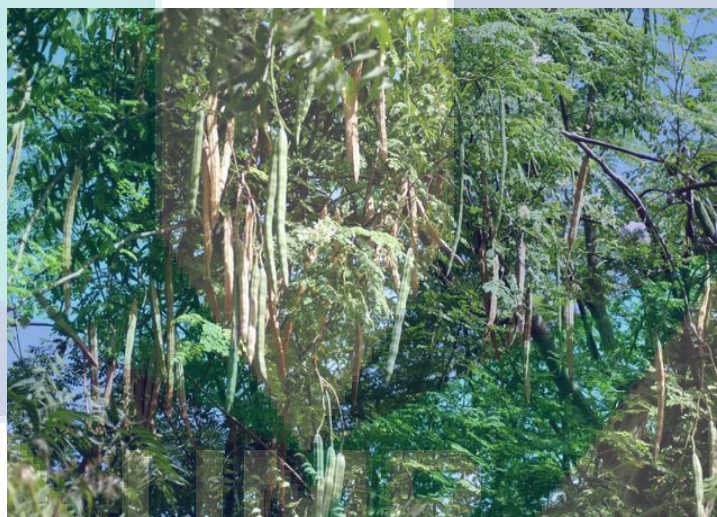


Figure 2.1: *Moringa oleifera* tree

The seed, leaf and pod of MO (Figure 2.2) are always used in medical field because of its rich nutritional profile and antioxidants galore (Mercola, 2015). Some research also proved that the seed, bark and leave of MO can used in water treatment to reduce the turbidity and heavy metal (Prasad and Rao, 2013; Alfarra et al., 2014). For example, its seeds have been used for few generation in Sudan and India for treatment of drinking water (Ravikumar and Sheeja, 2014). In addition, the leaves and fruits of the *Moringa oleifera* can be consumed or eaten as vegetables

(Madzvamuse, 2015). The leaves of the MO contain a lots of vitamins and minerals which had been to fight malnutrition used in India and Africa (WebMD, 2016).



Figure 2.2: *Moringa oleifera* seed, leaf and pod

Most of the part of *Moringa oleifera* can be used in medical treatment due to it high nutrient value. Besides, some of the part which contain active coagulant (polyelectrolyte) are commonly used as water purification (Yongabi, 2012). Table 2.1 show some of the uses from each part of the *Moringa oleifera*.

Table 2.1: Uses of various part from *Moringa oleifera*

Part	Uses	References
Seed	Water treatment Rheumatism treatment Treatment on Venomous Bite Abdominal Tumors Treatment	Madzvamuse, 2015; Anwar et al., 2007
Leave	Animal Fodder Vegetables Water Treatment Antitumor Sore throat treatment	Madzvamuse, 2015; Bodlund, 2013; Anwar et al., 2007
Press cake	Water purification Fertilizer	Madzvamuse, 2015; Alfarra et al., 2014
Fruit	Vegetable	Madzvamuse, 2015
Oil (Extraction from seed)	Cooking Soap manufacture Lubricant and Cosmetics production	Madzvamuse, 2015; Yongabi, 2012.
Roots	Pickle with vinegar Antilithic	Madzvamuse, 2015; Anwar et al., 2007

	Anti-inflammatory	
Bark	Water treatment Food seasoning	Alfarra et al., 2014; Madzvamuse, 2015
Wood	Heavy metal removal (act as active carbon)	Madzvamuse, 2015

2.2 Water Treatment with Some Natural Coagulant

There are some research had been done on the water treatment and heavy metal removal by using *Moringa oleifera* and some other natural coagulant. Below are some example of these research with method and result.

2.2.1. Removal of Metals Copper, Cadmium, Chromium, Zinc, And Lead Adsorbed By Moringa, Urad, Peanut, Bean And Corn

The Moringa, Urad, Peanut, Bean and Corn were obtained in the Fiji (Vikashni et al., 2012). The research was started by mixing 5 cm³ of known concentration of metal solution with 0.1 g of seed paste and left for 2 hours. *Moringa* showed more than 50 % removal for all tested metal. Other seeds did showed metal removal but with lower removal percentages. The summary of the removal percentage are shown in Figure 2.3.

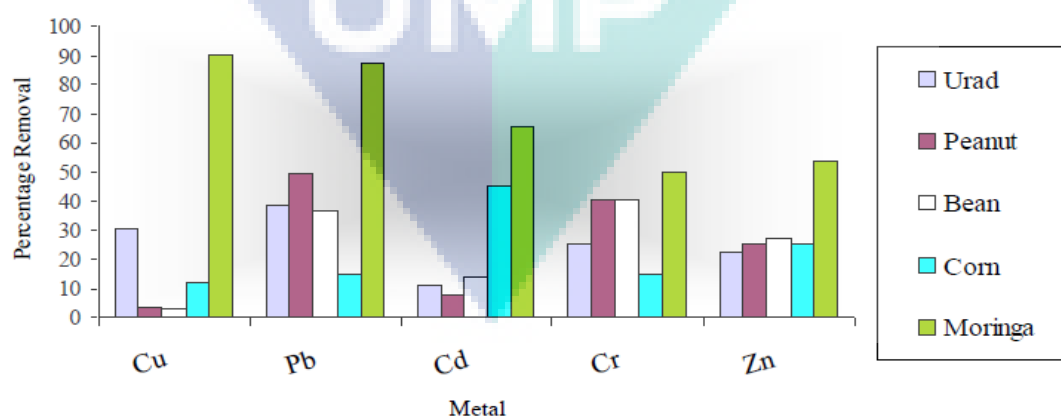


Figure 2.3: Percentage Removal of Heavy Metals Using *Moringa* and other Local Seeds

The adsorption of metals by *Moringa* is limited to the adsorption surface because of the cationic polyelectrolyte of short chain and low molecular weight (Muyibi et. al. 2002). The experiment show increase in removal of metals with the increasing dose of *Moringa* seeds coagulants.

2.2.2. Bioremediation of Heavy Metals using Leaves of *Calotropis procera*

The *Calotropis procera* was collected from National Institute of Technology, Karnataka of India (Patil et al., 2013). *Calotropis procera* were washed, oven-dried and crushed to sieved size fraction 0.5-1 mm with an analytical mill. Heavy metals of lead, copper, zinc, nickel were used. The experiments were conducted by mixing 50 ml of different concentration of heavy metal solutions with 0.1 g of *Calotropis procera* biomass (the crushed leaves) in the Erlenmeyer flasks. The flask were agitated at 150 rpm for 6 h before the metal solutions were separated from the biomass for analysis. Result showed that the metal removal percentage for lead, zinc, copper and nickel were 85.87%, 85.06%, 84.61% and 85.02% respectively. Higher initial concentration increased the driving force and enhanced the biosorption process until concentration reached 100 mg/l which might be the saturation of the biosorption sites on the biomass surface.

2.2.3. Sago for Heavy Metals and Turbidity Removal

Starch of sago obtained in Malaysia was prepared by peeling, crushing, filtering, washed and dried for 24 hours to form powder starch (Aziz et al., 2000). Metals (nickel, manganese, zinc, iron, and cadmium) were prepared with initial concentration 5 mg/L. The concentration of the metal in sample before and after jar test was measured with Shimadzu AAS to calculate the percentage removal. Under optimum pH (7.5) and Alum concentration (25 mg/L) the removal percentage of the metals iron increase form 10% removal to 30% removal and cadmium increase from 8% removal to 13% removal. This experiment showed sago as anionic polymer and would neutralize the positively metal ion.

2.2.4. Activated Carbon Prepared from Coconut Shell for Heavy Metal Removal

The coconut shell was obtained from fruits selling source, in Minna, Niger State (Bernard et al., 2013). Coconut shell was cleaned to remove any strange materials before dried and crushed to smaller size. Then, small piece of coconut shell was carbonized at 400 °C for 15 minutes and activated by 1.0M of ZnCl₂ at 500 °C for 3 hours. Next, it was washed until pH at range 6 to 7 and then dried again at 105 °C. 0.2 g activated carbon was added into 50 ml wastewater and stirred at 150 rpm for 2 hours. The optimum metal removal percentage were shown below.

Table 2.2: Metal Removal Percentage by Coconut Shell

Metal	Copper, Cu	Iron, Fe	Lead, Pb	Zinc, Zn
RP, (%)	71.26	76.02	100	26.15

Besides, research showed increase of both contact time, dosage, stirring speed and pH (maximum to pH 6) increased the metal removal percentage from the wastewater.

2.2.5. Wastewater treatment with *Tamarindus Indica* and *Moringa oleifera*

Moringa oleifera seed was deshelled and dried before milled to fine powder. The powder is then stored at 3 °C for 7 days. *Tamarindus Indica* was dried at 60 °C for 1 hour and grinded into powder form. The experiment was done with Jar test. The turbidity, hardness and metal concentration of the sample were measured by Nephelometer, complexometric titration and AAS respectively. The result of the experiment are shown in the Table 2.3. (Prasad and Rao, 2016)

Table 2.3: Result of water treatment by *Tamarindus Indica* and *Moringa oleifera*

Natural Coagulant	Metal Removal Percentage, %			Hardness Removal, %	Turbidity Reduced, NTU
	Cr	Cd	Zn		
<i>Tamarindus Indica</i>	62	73	70	10	0.5
<i>Moringa oleifera</i>	58	70	65	34	1.7

2.3 Water Treatment with *Moringa oleifera*

2.3.1. Comparison of Metal Removal with Whole Seed Kernel (WSK) and Ram Press Cake (RPC)

Dry *Moringa oleifera* seeds used in this study were obtained from Chikwawa District in Southern Malawi and identified by the Forestry Research Institute of Malawi (Sajidu et al., 2005). The seeds were deshelled and the kernel was powdered which was known as whole seed kernel (WSK). The cakes were the residues obtained after oil extraction from the seeds using a ram press and termed as Ram Press Cakes (RPC). The 1000 ppm of Pb, Fe, Cd samples solutions were prepared and diluted to give initial concentrations of 5.00 and 7.00 ppm. The jar test was conducted by mixing 100 mL of the diluted metal solutions with either WSK or RPC, with continuous stirring. Then the mixture was filtered and the metal concentration of the filtrate was measured by AAS. The results show the potential of *Moringa oleifera* in removal of iron, lead and cadmium were up to 92%, 89% and 48% respectively. The press cake remaining after oil extraction is richer in the active coagulants). (Henry, 2002). Hence, the optimum doses for metal ion removal for RPC were only 0.25 g/100ml compare to 2 g/100ml for WSK. Higher initial metal ion concentration required longer time for the complete removal.

2.3.2. Heavy Metal Removal in Aqueous Systems Using *Moringa oleifera* Seed

Moringa oleifera Lamarck seeds were collected in 2004 and the seeds were washed and dried at 65 °C (Kumari et al., 2006). *Moringa oleifera* is natural adsorbent which is not synthetically produced and has adsorptive properties. It has the ability to purify water especially in removal of heavy metals such as Ni, Cu, Zn, Pb, Cd, Cr, Ni, Cd and Zn. Recoveries of the metal with *Moringa oleifera* is high and up to 60.21% for As (III) and 85.60% for As (V), 76.59% for Cd (II), 68.85% for Cr (III) and 60.52% for Ni (II). Low concentrations of Cd in alcohol and Ag in water can be removed with these seeds after a pre-concentration step. Beside the seeds, the leaves can be applied in heavy metal sequestration studies, although in a lesser extent (Ndibewu et al., 2011).

2.3.3. Heavy Metal Removal from Water using *Moringa oleifera* Seed Coagulant and Double Filtration

Dry *Moringa oleifera* pods used in this research were collected from Varkala, Trivandrum (Ravikumar and Sheeja, 2013). Aqueous extract was prepared by stirring mixer of 200ml distilled water and 25 g MO seed powder for 1 hour before left for settling in 20 minutes. Jar tests were carried out by mixing the 2g/L of extract with 5mg/L of heavy metal solutions. Then, double filtration was used with an up flow roughing filtration and rapid filter as second stage to separate the solution with extract. The result showed that the removal percentage of copper, lead, cadmium and chromium were 95%, 93%, 76% and 70% respectively. However the turbidity had increased after the treatment and hence double filtration was used to reduce the turbidity of solutions below 2 NTU.

2.3.4. Comparison Metal Removal with *Moringa oleifera* Seed and Charcoal *Moringa oleifera* Seed

Moringa oleifera seeds were obtained from the Crop Science Department, University of Zimbabwe (Madzvamuse, 2015). Pretreatment was done by cleaning, drying and grounding the seed until powder before soxhlet extraction to remove oil. Some of the powder was then slowly heated until changed to charcoal. Known concentration of metal solutions and the charcoal *Moringa oleifera* seed (CMOS) or *Moringa oleifera* seed (MOS) were mixed and shaken. Finally concentration of unabsorbed metal ions were determined. The result showed both CMOS and MOS effectively remove chromium, lead and cadmium at low initial concentration of metals. The CMOS and MOS were successfully remove 46% and 67% of cadmium respectively. When the concentration of metal increase, the efficiency of metal removal decrease for both CMOS and MOS. The cadmium removal percentage increased from 6% to 59% when the dosage of MOS used increased from 0.5g to 1.5g. CMOS did not show increase in removal percentage of cadmium when the dosage increased.

2.3.5. Water Treatment with *Moringa oleifera* Leaves

Moringa oleifera leaves were collected from nearby area of Universiti Malaysia Pahang, Kuantan, Pahang, Malaysia (Ali et al., 2015). *Moringa oleifera* leaves were collected, dried and grounded to different particle sizes. Kaolin and sodium bicarbonate were used to prepared solution with different turbidity and different concentration of Cd (II) solutions were prepared by diluting stock solution. Equipment used in this experiment were AAS 400, pH meter and turbidity meter. The results showed that the removal percentage (RP) of 1ppm of Cd (II) solution increased from 68% to 72% when dosage of MO leaves increased from 1g/L to 6g/L. When the contact time increased, the RP of Cd (II) kept on increasing to 71% at 18 minutes. Smaller size of absorbance also increase the RP where 81% of RP was achieved when the particles size were smaller than 250 μm . There were no significant effect of pH on the RP. Increased in turbidity will decrease the RP.

2.3.6. *Moringa Oleifera* Leaves Powder in Removing Mn, Ni, Cr, Cu, Fe and Zn

Moringa oleifera was provided from native shrubs of Bushehr province, south of Iran *Moringa oleifera* was provided from native shrubs of Bushehr province, south of Iran (Farrokhzadeh et al., 2013). *Moringa oleifera* was powdered to different size after cleaning and drying. 50, 100 and 200 mg/L of metal solutions were diluted from stock solutions. The MO powder was added to 100ml of metal solution and stirred (350 rpm) for 1 hour and settled for 30 minutes. The solution was then filtered and analyzed. The signal-to-noise (S/N) was higher when the initial metal concentration was 50 mg/L which indicate the lowest absorption of metal ions. The optimum pH for removal of Fe and Cu were 7, Mn at pH 5 while other metals at pH 3. The removal percentage also ascended with the increasing of dosage and particle size. The results were different from other research where lower particles size gained higher removal percentage which might due to very small particles size released back the metal during filtration.

CHAPTER 3

METHODOLOGY AND METHODS

3.1 Materials and Apparatus

- Cu, Cr, and Fe standard solution with concentration of 1000 mg/L from PerkinElmer.
- Wastewater samples from Balok River, Gebeng Kuantan.
- *Moringa oleifera* press cake from MitoMasa Sdn Bhd, Kuala Lumpur
- Glassware
- Vacuum filtration apparatus
- Atomic Absorption Spectrophotometer
- Turbidity Meter
- pH meter
- BOD incubator
- Dissolved oxygen meter for BOD
- Spectrophotometer, HACH DR/2400 @ 2800 for COD

3.2 Methodology

3.2.1 Pre-treatment on MO Press Cake

The MOPC was immersed into distilled water for 24 hours to remove the remaining oil. Then the MOPC was then filtered and dried in the chiller for 1 day. However the moisture was not totally removed, so approximate 0.6 g of the MOPC was heated to 100 °C for 24 hours. Then, the dry mass of the MOPC was measured to calculate the

remaining moisture content in the MOPC. Direct 100 °C heating will destroy the MOPC.

3.2.2 Metal Stock Solution Preparation

Each type of metal solution was first diluted to concentration of 10 mg/L in 100 mL of volumetric flask from standard stock solution (1000 mg/L) with diluted water. Then the stock solution were further diluted in 50 mL volumetric flask with concentration of:

- i. 0 mg/L, 1 mg/L, 2 mg/L and 4 mg/L of iron solution.
- ii. 0 mg/L, 1 mg/L, 2 mg/L and 4 mg/L of copper solution.
- iii. 0 mg/L, 1 mg/L, 3 mg/L and 5 mg/L of chromium solution.

These solution were used to prepare standard for calibration curve. A drop of nitric acid was added to the solution for preservation (Vikashni et al., 2012).

3.2.3 *Moringa Oleifera* Solution Preparation

The MO solution was prepared by adding 10 g (dry mass) of MO into 500 mL of distilled water to make a 20,000 ppm MO solution. The solution was stirred so the MO can be dissolved in the dilution water. Then the solution was further diluted to 10 mL of 1,000 ppm, 5,000 ppm, 10,000 ppm, 15,000 ppm and 20,000 ppm. For example, to prepare 10 mL of 1,000 ppm of *Moringa oleifera* solution, using equation (3.5), 0.5 mL of 20,000 ppm solution was diluted by 9.5 mL of distilled water.

3.2.4 Wastewater Treatment

Each beaker in jar test was filled with 500 mL of solution, one beaker was used as blank and other beakers were filled with wastewater. These six beaker were placed into the jar test machine. The 10 mL of MOPC with different concentration prepared were added into different sample solution. The stirring paddles was lowered and immersed into the solution. The stirring speed was set as 200 rpm for 4 minutes and 40 rpm for 30 minutes (Lea, 2010).

3.2.5 Heavy metal measurement

The heavy metal removal test was done by using Polarized Zeeman Atomic Absorption Spectrophotometer (Model Z-5000 Series). Calibration curve for each type of metal was obtained from the stock solution prepared from each standard at different concentration. The wastewaters obtained after the jar test were filtered by using vacuum pump through a 0.45 μ m nylon membrane filter before tested by AAS (Ali et al., 2014).

3.2.6 Turbidity and pH Measurement

The turbidity and pH of wastewater sample were be measured by turbidity meter and pH meter respectively.

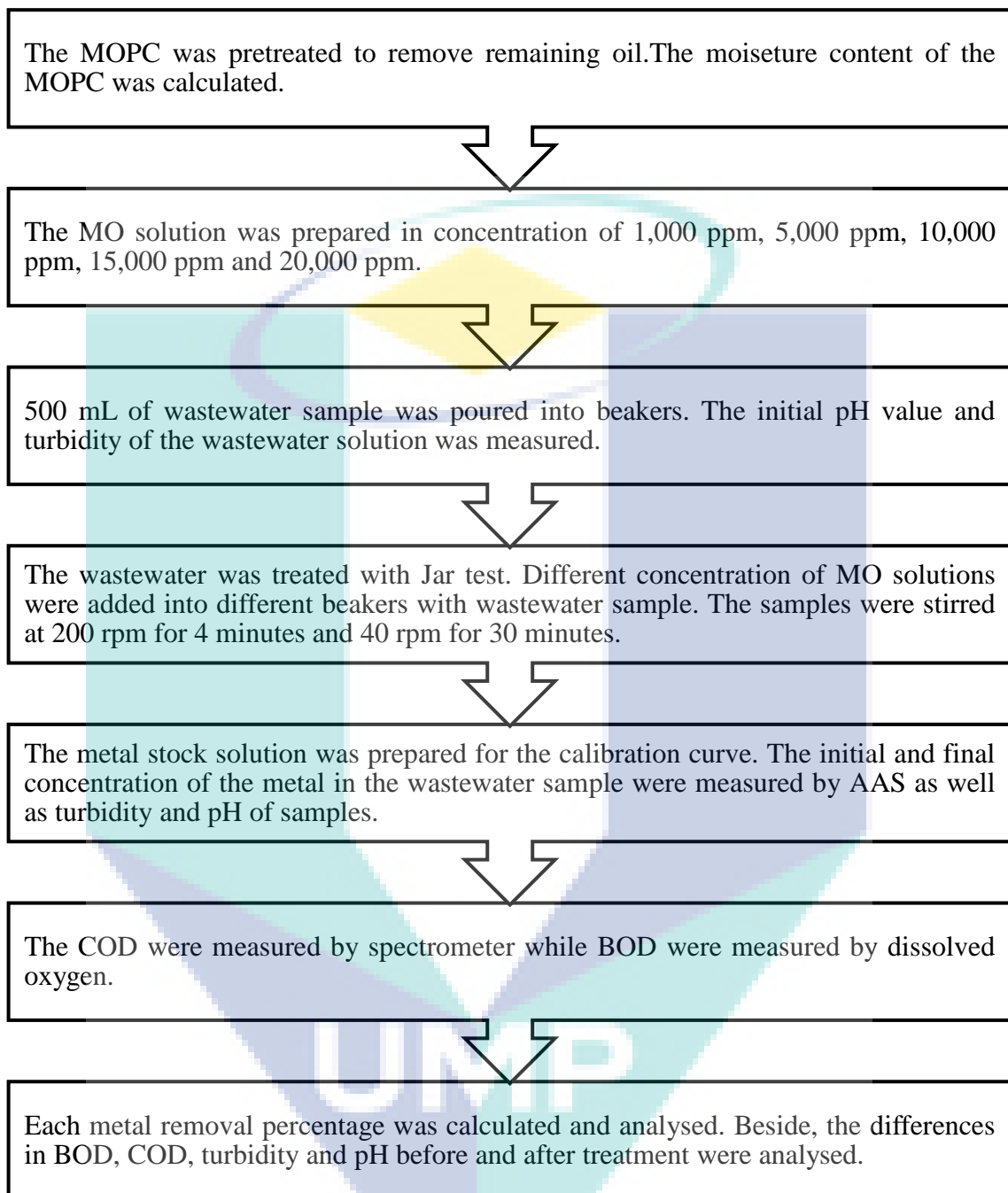
3.2.7 Biochemical Oxygen Demand (BOD) Measurement

BOD measurement was done by dissolved oxygen meter. Dilution water was prepared by mixing 3 mL of phosphate buffer, magnesium sulfate, calcium chloride, ferric chloride solution into 3 L volumetric flask. 10 mL of samples after jar test were mixed with 300 mL of dilution water into incubation bottle. DO was measured for all the samples. Water was added to the flared mouth of bottle and covered with an aluminum foil. All the bottles were then put into BOD incubator for 5 days at 20 °C. The final DO was measured and BOD of each sample was calculated by equation (3-3). (Suhartini et al., 2013)

3.2.8 Chemical Oxygen Demand (COD) Measurement

Spectrophotometer was used to measure the COD. 2 mL of de-ionized water (control) and samples after jar test were added into COD Digestion Reagent Vials. Then, the samples were inserted into COD reactor and heated for 2 hours at 150 °C with strong oxidizing agent (potassium dichromate solution). Then the solutions were cooled down to room temperature. The vials were cleaned to remove any finger print before the COD was measured with spectrophotometer. (Kasmawati and Lee, 2007)

3.2.9 Simple Flow Chart



3.3 Calculations

Moisture content (wet basis), % calculation:

$$MC = \frac{W_{wet} - W_{dry}}{W_{wet}} (100\%) \quad (3.1)$$

Wet basis dosage of MOPC calculation

$$W_{dry} = \frac{W_{wet}(100 - MC)}{100\%} \quad (3.2)$$

BOD calculation:

$$BOD_5 = \frac{D_i - D_f}{P} \quad (3.3)$$

Metal Removal Percentage, %:

$$RP = \frac{C_i - C_f}{C_i}(100\%) \quad (3.4)$$

Mole Balance:

$$M_1V_1 = M_2V_2 \quad (3.5)$$

Where:

C_i – Initial Metal Concentration (before treatment), mg/L

C_f – Final Metal Concentration (after treatment), mg/L

D_f – Final Dissolved Oxygen, mg/L

D_i – Initial Dissolved Oxygen, mg/L

M_1 – Initial molarity of solution, ppm

M_2 – Desired molarity of the solution, ppm

P - Decimal volumetric fraction of sample used, V_s/V_t

V_1 – Volume of concentrated solution needed, mL

V_2 – Desired volume of solution at desired molarity, mL

V_s - Volume of sample solution, mL

V_t - Volume of blank solution, mL

W_{dry} – Weight of dry *Moringa oleifera* Press Cake (After heating to 100°C) (g).

W_{wet} - Weight of wet *Moringa oleifera* Press Cake (g).

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Moisture Content

Since the dry mass was needed in the preparation of MO solution with different concentration. However the MO was not dried in 100 °C and the moisture content in the MO was not zero. Hence, the moisture content was calculated by using the equation (3.1), the moisture content of the MO is 39.70%.

With the moisture content calculated and the equation (3.2), 10 g of dry basis MO was equal to 16.5837 g of wet basis MO.

4.2 Heavy Metal Removal Percentage

The concentration of heavy metal of Fe and Cr metal in the wastewater of the Balok River Gebeng, Kuantan were approximately to 1 mg/L while Cu concentration was approximately 0.4 mg/L. To determine the optimum dosage or concentration of *Moringa oleifera* solution added into the wastewater, a jar test was done by adding 10 mL of 1000 ppm, 5000 ppm, 10000 ppm, 15000 ppm and 20000 ppm into 500 mL of wastewater. Table 4.1 and Figure 4.1 show the result of the heavy metal concentration of the wastewater from Balok River after the treatment with different concentration of *Moringa oleifera* solution.

Table 4.1: Metal concentration after Jar test

MO concentration, ppm	0	1000	5000	10000	15000	20000
Fe, ppm	0.803	0.376	0.332	0.241	0.308	0.388
Cu, ppm	0.395	0.128	0.044	0.118	0.102	0.097
Cr, ppm	0.734	0.212	0.072	0.058	0.046	0.051

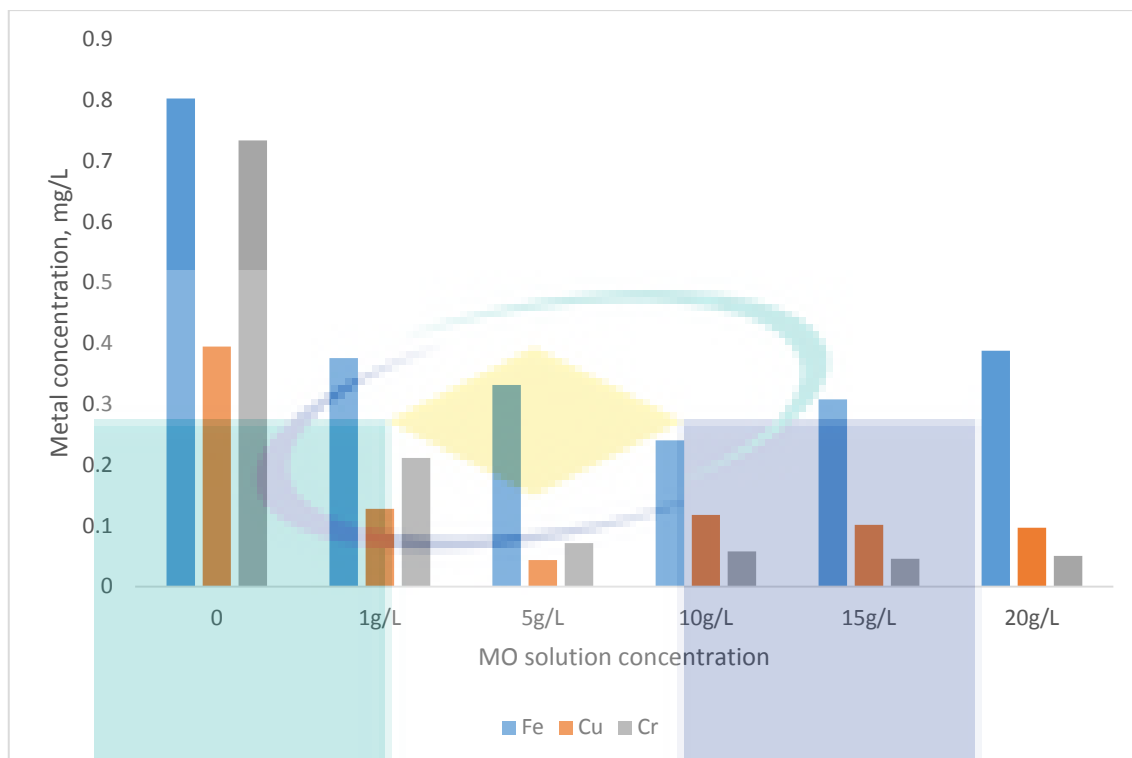


Figure 4.1: Metal Concentration after Jar test

The removal percentage of each type of metal at different concentration of MO solution were calculated with equation (3.4). The result of the removal percentage are shown in Table 4.2 and Figure 4.2.

Table 4.2: Metal Removal Percentage

MO concentration, ppm	0	1000	5000	10000	15000	20000
Fe, %	0	53.18	58.66	69.99	61.64	51.68
Cu, %	0	67.59	88.86	70.13	74.18	75.44
Cr, %	0	71.12	90.19	92.10	93.73	93.05

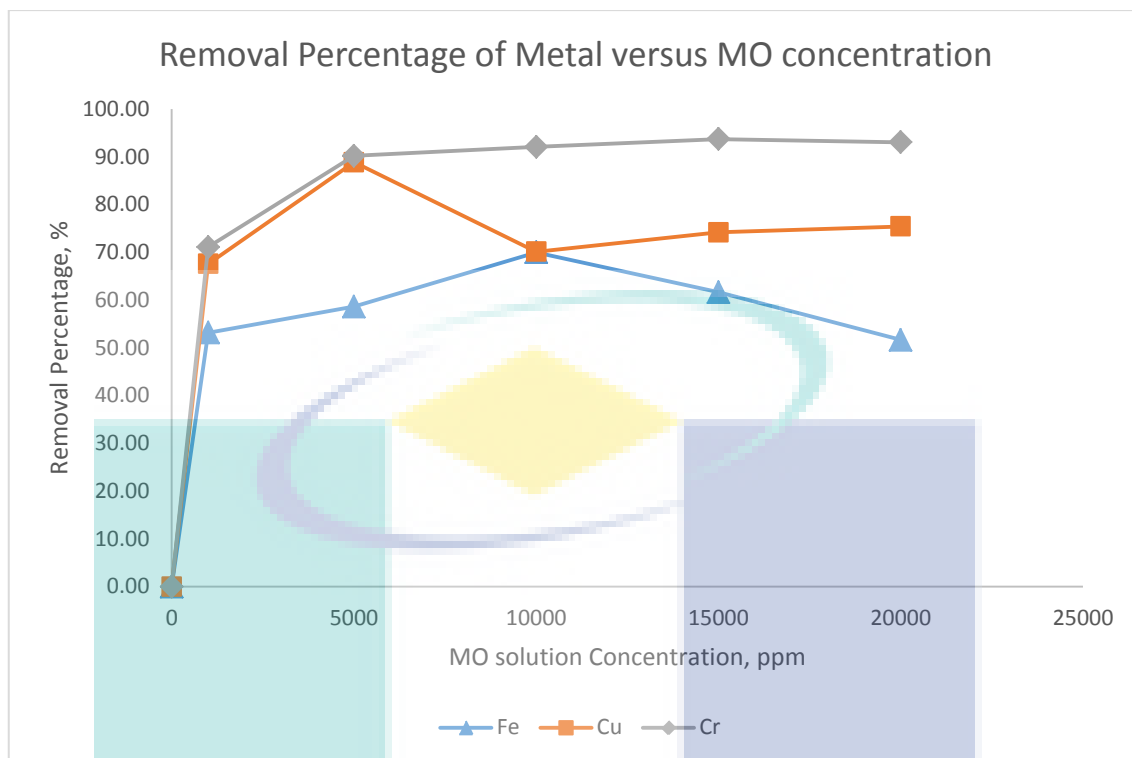


Figure 4.2: Metal Removal Percentages

From the result of the experiment, the heavy metal were successfully to be reduced after the treatment with the MOPC. The removal percentage of Iron, copper and chromium reached 69.99%, 88.86% and 93.73% respectively. Generally, the metal removal percentage increase proportional to the concentration of MO solution until an optimum concentration. Besides, the optimum MO concentration for iron, copper and chromium removal were at 10000 mg/L, 5000 mg/L and 15000 mg/L respectively. The removal of iron in this experiment were lower than the research done by Sajidu et al. (2005) which had a removal of 92.14 %. The removal percentage of chromium in this research was higher compared with the research done by Vikashni et al. (2012) which was only 60% and Ravikumar and Sheeja (2013) which was 73%. The copper removal efficiency in this research is approximately same to the literature statement of Vikashni et al. (2012) which was approximately to 90%. The trend of the removal percentage of copper and chromium were found to be alike as the trend in the research done by Ali (2016) and Mataka et al. (2010).

4.3 Turbidity, pH, COD, and BOD

By using the same methodology in processing the wastewater in determining the heavy metal removal, other parameters such as turbidity, pH, COD and BOD values were determined by different equipment.

4.3.1 Turbidity

The turbidity of the wastewater were determined by using the turbidity meter and the result were shown in the Figure 4.3.

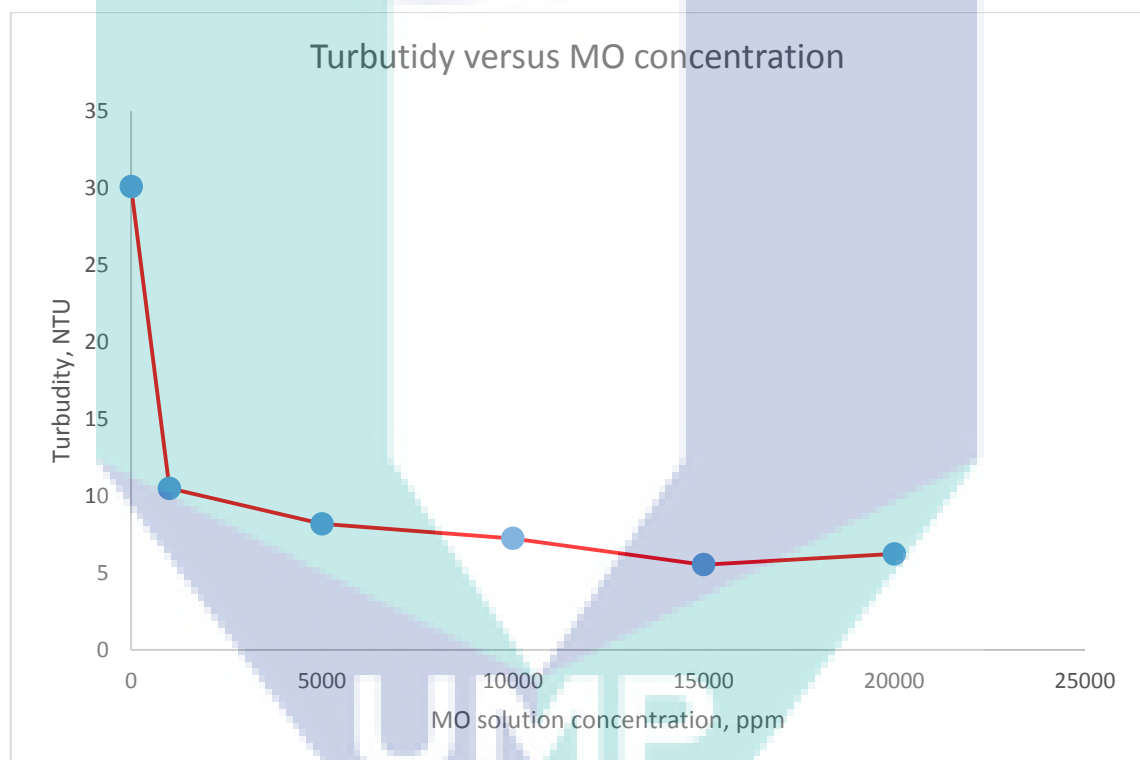


Figure 4.3: Turbidity of wastewater

From the result obtained in the experiment, the wastewater collected were having turbidity of 30.1 NTU. After the jar test, the turbidity were decrease to a minimum value of 5.54 NTU at the optimum concentration of 15000 ppm MO solution. From Figure 3.3, it had shown a trend of decreasing in turbidity when the MO solution concentration increase. However, there are a limitation for the turbidity removal and the continue increase of the MO will lead to increase residual turbidity (Katayon et al., 2007). The highest removal percentage of the turbidity was 81.6%.

4.3.2 pH

The pH value will be calculated by using the pH meter and the result are shown in the Figure 4.4.

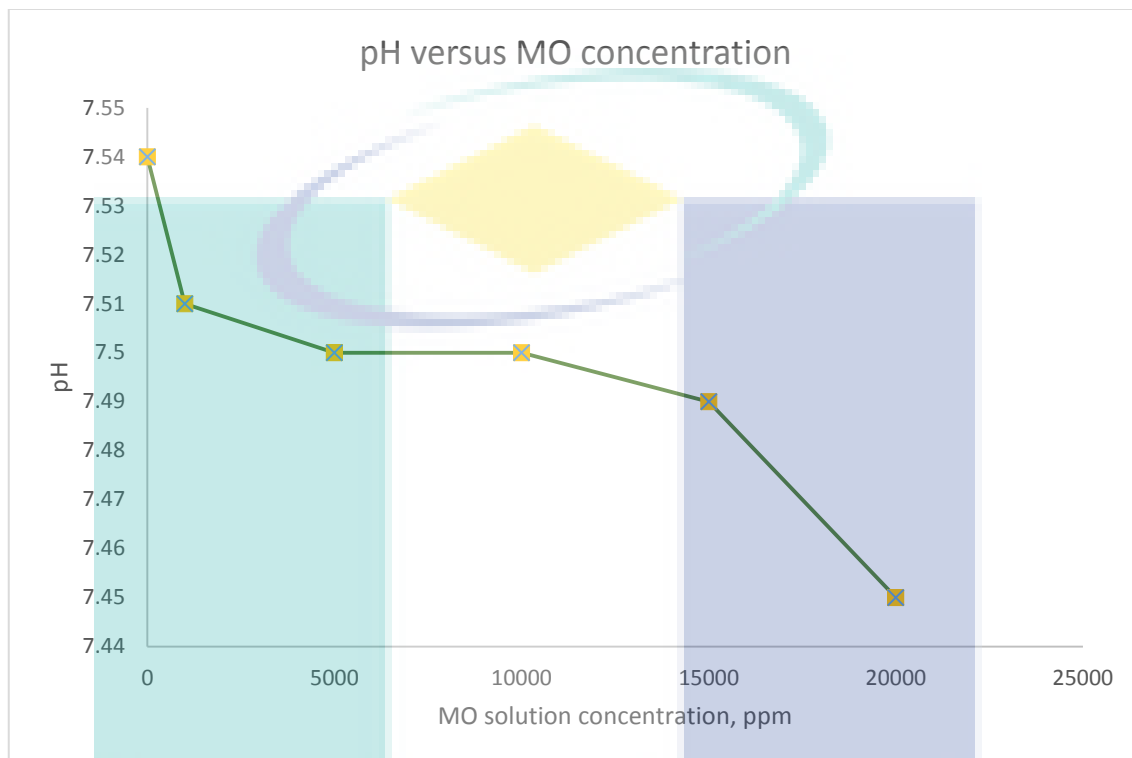


Figure 4.4: pH value of the wastewater

From the Figure 3.4, there was no significant differences in the pH value which is between 7.45 and 7.54 after treatment with different concentration of MOPC. The maximum differences after the treatment was only with a value of 0.09. This range is under the World Health Organization permissible limits of 6.5 to 8.5 for drinking water.

4.3.3 COD

The COD of the wastewater were determined by using the HACH DR/2400 @ 2800 after heated for 2 hours at 150 °C with strong oxidizing agent (potassium dichromate solution) in COD Digestion Reagent Vials. The result of COD were shown in the Figure 3.5.

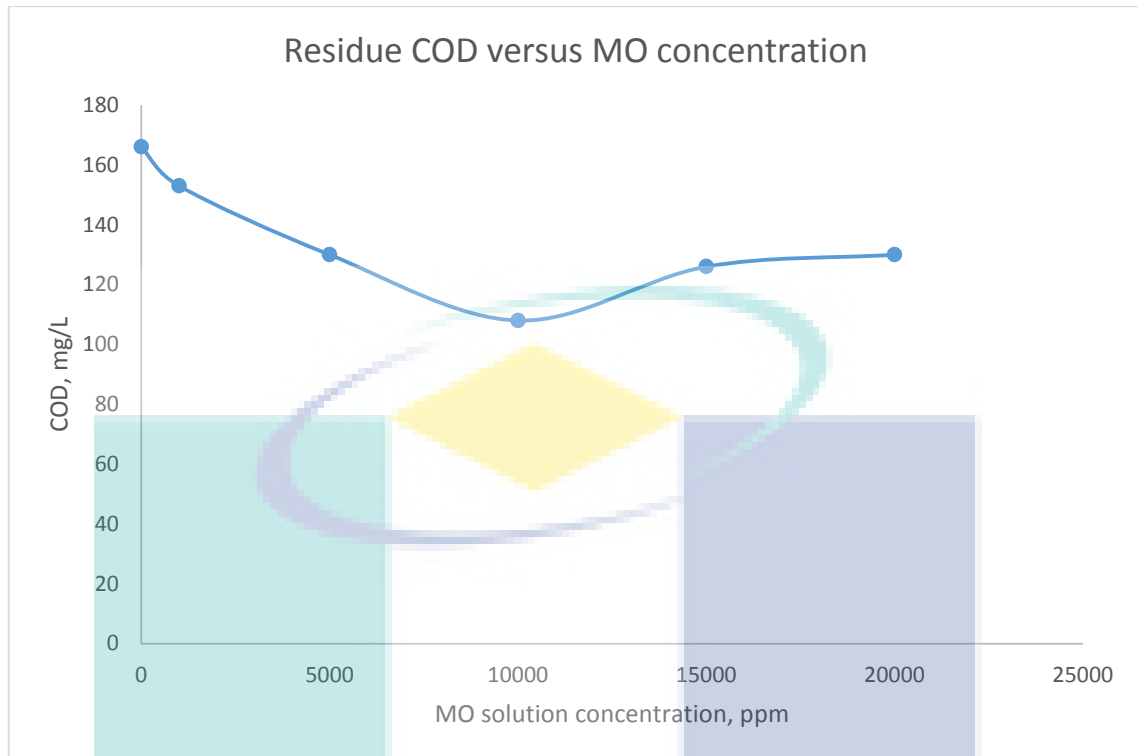


Figure 4.5: COD of the wastewater

From the Figure 4.5, the MO did shown the usage of reducing the COD of the wastewater. The COD of the wastewater decrease from 166 mg/L to 108 mg/L at the optimum concentration of 10000 ppm. Then the COD increase slightly with the increase of MO solution concentration after it reached the optimum concentration. The maximum COD removal percentage was 34.94 %. The result had shown similar phenomenon with previous research where there are an optimum dosage of MO and continue increase of MO dosage will increase the COD (Hemapriya et al., 2015).

4.3.4 BOD

The DO of the wastewater initially and after 5 days were determined by using the dissolved oxygen. Then, the BOD of each sample was calculated by equation (3.3) and the result are shown in the Table 4.3 and Figure 4.6.

Table 4.3: DO and BOD of the wastewater

MO solution	0	1000	5000	10000	15000	20000

concentration, ppm						
DO _i , mg/L	1.31	1.30	1.32	1.30	1.29	1.29
DO _f , mg/L	1.01	0.98	0.84	0.79	0.73	0.68
BOD, mg/L	9.0	9.6	14.4	15.3	16.8	18.3

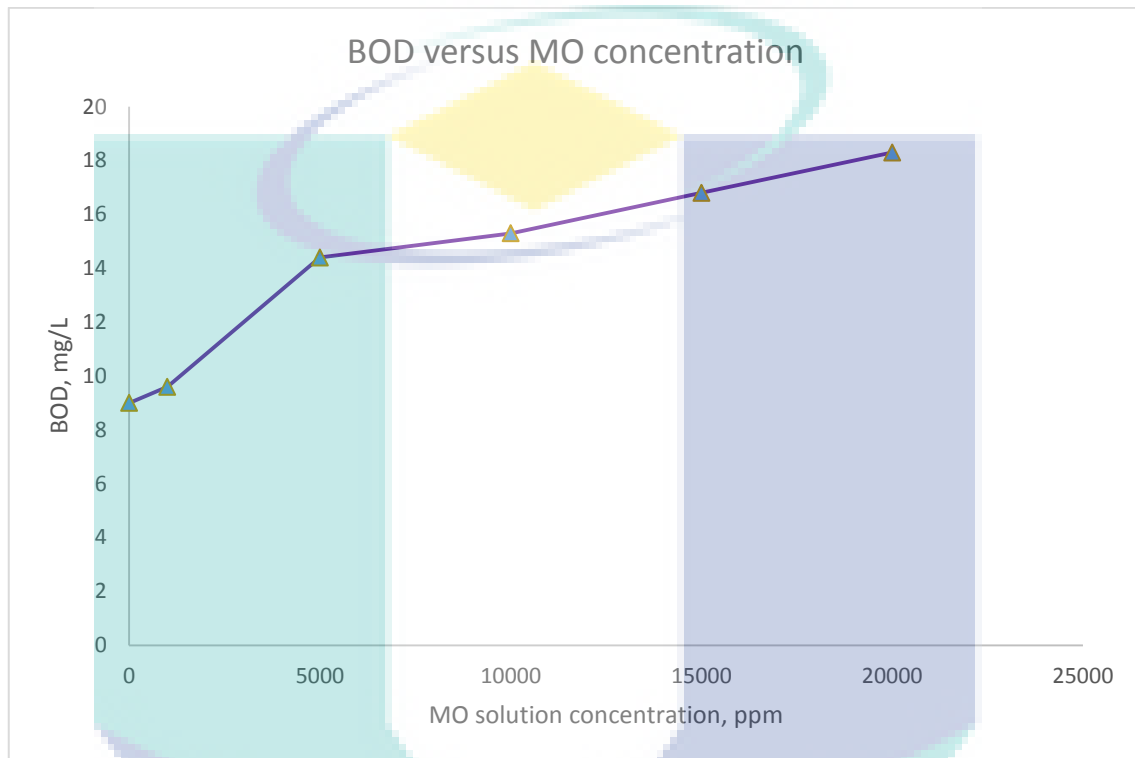


Figure 4.6: BOD of the wastewater

According to the result, the DO reduced from 1.31 mg/L to 0.68 mg/L and the BOD value increase as the increase of the MO solution concentration. The BOD had increase from 9 mg/L to 18.3 mg/L when 25000 ppm of the MO solution was added to the wastewater. The result are different from the previous research which had shown the removal of the BOD by the *Moringa oleifera*. The increase in BOD indicated the increase of the oxygen demand when higher concentration of the MO solution was added to the sample. This is due organism compounds in the MOPC enable the growth of good organism in the water. The decreased in DO value might be caused by the increase in oxygen needed by MO to oxidize the organic substance inside the wastewater (Yuliastri et al., 2016).

CHAPTER 5

CONCLUSION AND RECOMMENDATION

In a nutshell of this research, the MOPC had been proved to remove 69.99% Fe, 88.86 % Cu and 93.73% Cr at optimum concentration of 10000 ppm, 5000 ppm and 15000 ppm respectively. The increase in the MO concentration increased the heavy metal removal percentage until the optimum concentration was used. Moreover, the turbidity also had been reduced as much as 81.6% with only changes of 0.09 pH value. Meanwhile, MO can reduce the 34.94 % of COD but increase the BOD from 9.0 ppm to 18.3 ppm. The result had shown that the MOPC are able to remove both Fe, Cu and Cr metal from wastewater.

For the recommendation of this study, the *Moringa oleifera* press cake should not be stored in chiller more than 1 days to avoid the growing of the organisms which may affect the result obtained. Besides, the filtered samples should be tested directly without storing because it might affect the result. Future research on the heavy metal removal by *Moringa oleifera* press cake at different pH condition can be done.

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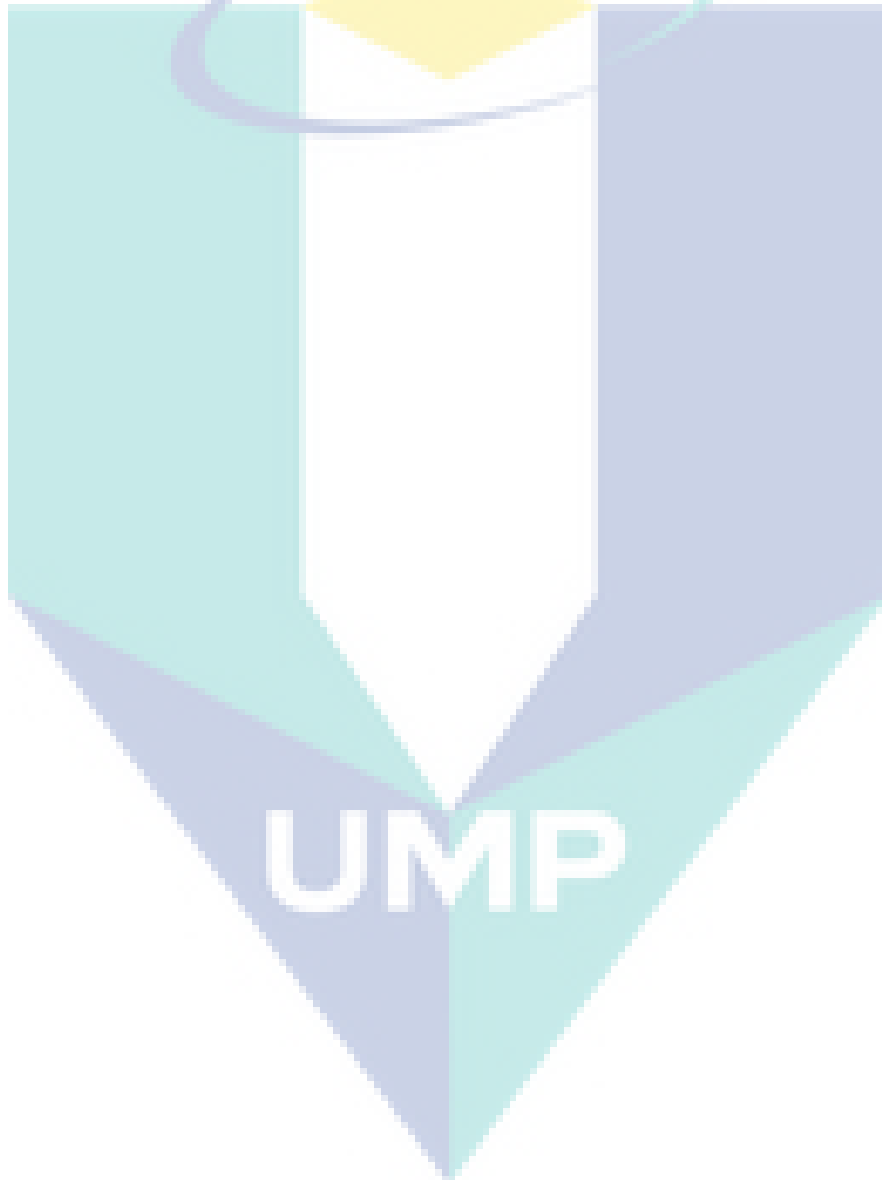
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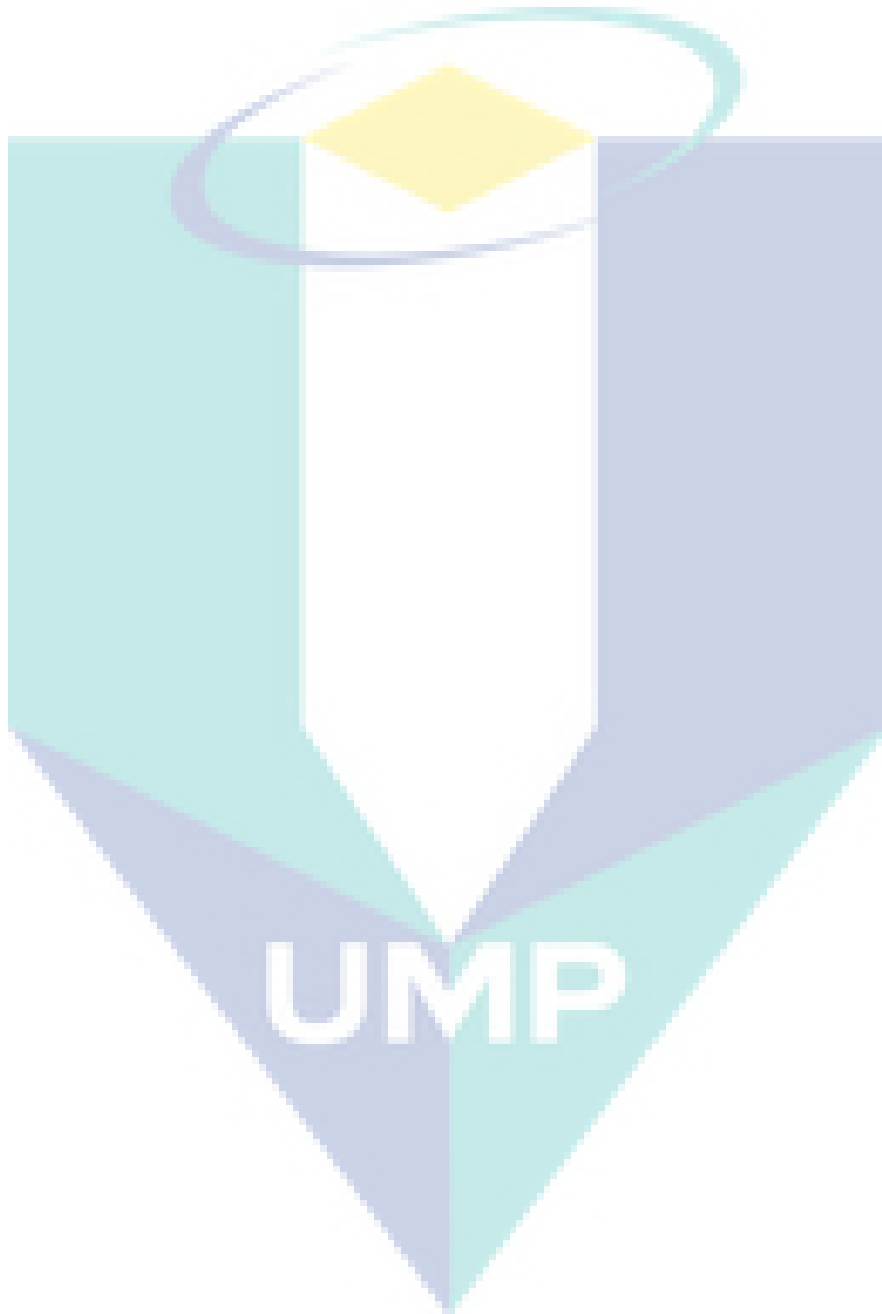
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APPENDICES



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Page 1

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Batch ID:

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Analyte: Cr 357.87

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2	[0.00]	0.198	0.198	10:04:13 PM	Yes
3	[0.00]	0.198	0.198	10:04:18 PM	Yes
Mean:	[0.00]	0.197			
SD:	0.0000	0.0014			
%RSD:	0.00%	0.70			

Auto-zero performed.

Sequence No.: 2

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2	[1]	0.032	0.032	10:04:55 PM	Yes
3	[1]	0.033	0.033	10:05:00 PM	Yes
Mean:	[1]	0.032			
SD:	0.00	0.0002			
%RSD:	0.00%	0.55			

Standard number 1 applied. [1]

Correlation Coef.: 1.000000 Slope: 0.03244 Intercept: 0.00000

Sequence No.: 3

Autosampler Location:

Sample ID: Calib Std 2

Date Collected: 11/24/2011 10:05:28 PM

Analyst:

Data Type: Original

Replicate Data: Calib Std 2

Analyte: Cr 357.87

Repl #	SampleConc mg/L	StndConc mg/L	Blncorr Signal	Time	Signal Stored
1	[3]	0.081	0.081	10:05:29 PM	Yes
2	[3]	0.084	0.084	10:05:33 PM	Yes
3	[3]	0.083	0.083	10:05:38 PM	Yes
Mean:	[3]	0.083			
SD:	0.00	0.0013			
%RSD:	0.00%	1.62			

Standard number 2 applied. [3]

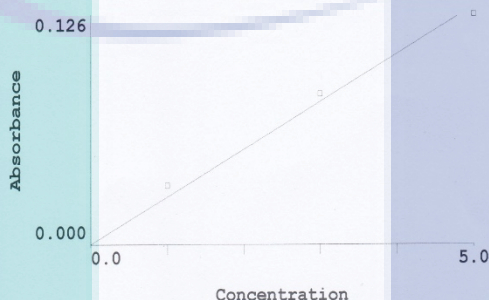
Correlation Coef.: 0.993064 Slope: 0.02809 Intercept: 0.00000

Sequence No.: 4 Autosampler Location:
 Sample ID: Calib Std 3 Date Collected: 11/24/2011 10:06:03 PM
 Analyst: Data Type: Original

 Replicate Data: Calib Std 3 Analyte: Cr 357.87

Repl #	SampleConc mg/L	StndConc mg/L	Blncorr Signal	Time	Signal Stored
1	[5]	[5]	0.125	10:06:04 PM	Yes
2	[5]	[5]	0.127	10:06:09 PM	Yes
3	[5]	[5]	0.127	10:06:13 PM	Yes
Mean:	[5]	[5]	0.126		
SD:		0.00	0.0008		
%RSD:		0.00%	0.63		

Standard number 3 applied. [5]
 Correlation Coef.: 0.993010 Slope: 0.02613 Intercept: 0.00000
 The calibration curve may not be linear.



 Calibration data for Cr 357.87 Equation: Linear Through Zero

ID	Mean Signal (Abs)	Entered Conc. mg/L	Calculated Conc. mg/L	Standard Deviation	%RSD
Blank	0.000	0	0.000	0.00	0.70
Calib Std 1	0.032	1.0	1.241	0.00	0.55
Calib Std 2	0.083	3.0	3.160	0.00	1.62
Calib Std 3	0.126	5.0	4.834	0.00	0.63

Correlation Coef.: 0.993010 Slope: 0.02613 Intercept: 0.00000

 Sequence No.: 5 Autosampler Location:
 Sample ID: 0 Date Collected: 11/24/2011 10:06:43 PM
 Analyst: Data Type: Original

 Replicate Data: 0 Analyte: Cr 357.87

Repl #	SampleConc mg/L	StndConc mg/L	Blncorr Signal	Time	Signal Stored
1	0.808	0.808	0.021	10:06:44 PM	Yes
2	0.727	0.727	0.019	10:06:49 PM	Yes
3	0.667	0.667	0.017	10:06:53 PM	Yes
Mean:	0.734	0.734	0.019		
SD:	0.0707	0.0707	0.0018		
%RSD:	9.64%	9.64%	9.64		

 Sequence No.: 6 Autosampler Location:
 Sample ID: 1 Date Collected: 11/24/2011 10:07:24 PM
 Analyst: Data Type: Original

 Replicate Data: 1 Analyte: Cr 357.87

Repl #	SampleConc mg/L	StndConc mg/L	Blncorr Signal	Time	Signal Stored
1	0.268	0.268	0.007	10:07:25 PM	Yes
2	0.207	0.207	0.005	10:07:30 PM	Yes
3	0.161	0.161	0.004	10:07:34 PM	Yes

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Date: 11/24/2011 10:11:10 PM

Mean: 0.212 0.212 0.006
 SD: 0.0541 0.0541 0.0014
 %RSD: 25.49% 25.49% 25.49

Sequence No.: 7
 Sample ID: 5
 Analyst:

Autosampler Location:
 Date Collected: 11/24/2011 10:08:05 PM
 Data Type: Original

Replicate Data: 5 Analyte: Cr 357.87

Repl #	SampleConc mg/L	StndConc mg/L	Blncorr Signal	Time	Signal Stored
1	0.086	0.086	0.002	10:08:07 PM	Yes
2	0.034	0.034	0.001	10:08:11 PM	Yes
3	0.096	0.096	0.003	10:08:15 PM	Yes
Mean:	0.072	0.072	0.002		
SD:	0.0335	0.0335	0.0009		
%RSD:	46.49%	46.49%	46.49		

Sequence No.: 8
 Sample ID: 10
 Analyst:

Autosampler Location:
 Date Collected: 11/24/2011 10:08:46 PM
 Data Type: Original

Replicate Data: 10 Analyte: Cr 357.87

Repl #	SampleConc mg/L	StndConc mg/L	Blncorr Signal	Time	Signal Stored
1	0.076	0.076	0.002	10:08:48 PM	Yes
2	0.059	0.059	0.002	10:08:52 PM	Yes
3	0.040	0.040	0.001	10:08:57 PM	Yes
Mean:	0.058	0.058	0.002		
SD:	0.0178	0.0178	0.0005		
%RSD:	30.48%	30.48%	30.48		

Sequence No.: 9
 Sample ID: 15
 Analyst:

Autosampler Location:
 Date Collected: 11/24/2011 10:09:26 PM
 Data Type: Original

Replicate Data: 15 Analyte: Cr 357.87

Repl #	SampleConc mg/L	StndConc mg/L	Blncorr Signal	Time	Signal Stored
1	0.053	0.053	0.001	10:09:28 PM	Yes
2	0.037	0.037	0.001	10:09:32 PM	Yes
3	0.047	0.047	0.001	10:09:36 PM	Yes
Mean:	0.046	0.046	0.001		
SD:	0.0079	0.0079	0.0002		
%RSD:	17.19%	17.19%	17.19		

Sequence No.: 10
 Sample ID: 20
 Analyst:

Autosampler Location:
 Date Collected: 11/24/2011 10:10:05 PM
 Data Type: Original

Replicate Data: 20 Analyte: Cr 357.87

Repl #	SampleConc mg/L	StndConc mg/L	Blncorr Signal	Time	Signal Stored
1	0.069	0.069	0.002	10:10:07 PM	Yes
2	0.074	0.074	0.002	10:10:11 PM	Yes
3	0.009	0.009	0.000	10:10:15 PM	Yes
Mean:	0.051	0.051	0.001		
SD:	0.0363	0.0363	0.0009		
%RSD:	71.39%	71.39%	71.39		

Sequence No.: 16
 Sample ID: Blank
 Analyst:

Autosampler Location:
 Date Collected: 11/24/2011 9:51:46 PM
 Data Type: Original

Replicate Data: Blank Analyte: Fe 248.33

Repl #	SampleConc mg/L	StdConc mg/L	Blncorr Signal	Time	Signal Stored
1	[0.00]	[0.00]	0.418	9:51:47 PM	Yes
2	[0.00]	[0.00]	0.415	9:51:52 PM	Yes
3	[0.00]	[0.00]	0.416	9:51:56 PM	Yes
Mean:	[0.00]	[0.00]	0.416		
SD:	0.0000	0.0000	0.0015		
%RSD:	0.00%	0.00%	0.35		

Auto-zero performed.

Sequence No.: 17
 Sample ID: Calib Std 1
 Analyst:

Autosampler Location:
 Date Collected: 11/24/2011 9:52:19 PM
 Data Type: Original

Replicate Data: Calib Std 1 Analyte: Fe 248.33

Repl #	SampleConc mg/L	StdConc mg/L	Blncorr Signal	Time	Signal Stored
1	[1]	[1]	0.016	9:52:21 PM	Yes
2	[1]	[1]	0.006	9:52:25 PM	Yes
3	[1]	[1]	0.014	9:52:29 PM	Yes
Mean:	[1]	[1]	0.012		
SD:	0.00	0.00	0.0050		
%RSD:	0.00%	0.00%	41.13		

Standard number 1 applied. [1]
 Correlation Coef.: 1.000000 Slope: 0.01211 Intercept: 0.00000

Sequence No.: 18
 Sample ID: Calib Std 2
 Analyst:

Autosampler Location:
 Date Collected: 11/24/2011 9:52:55 PM
 Data Type: Original

Replicate Data: Calib Std 2 Analyte: Fe 248.33

Repl #	SampleConc mg/L	StdConc mg/L	Blncorr Signal	Time	Signal Stored
1	[2]	[2]	0.020	9:52:56 PM	Yes
2	[2]	[2]	0.023	9:53:01 PM	Yes
3	[2]	[2]	0.016	9:53:05 PM	Yes
Mean:	[2]	[2]	0.020		
SD:	0.00	0.00	0.0039		
%RSD:	0.00%	0.00%	19.61		

Standard number 2 applied. [2]
 Correlation Coef.: 0.961665 Slope: 0.01039 Intercept: 0.00000

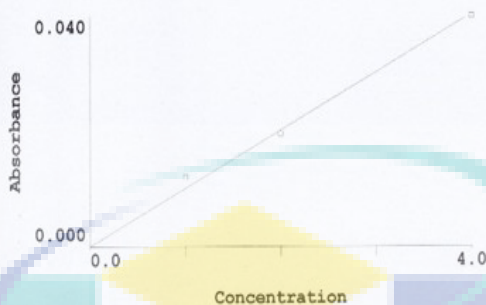
Sequence No.: 19
 Sample ID: Calib Std 3
 Analyst:

Autosampler Location:
 Date Collected: 11/24/2011 9:53:33 PM
 Data Type: Original

Replicate Data: Calib Std 3 Analyte: Fe 248.33

Repl #	SampleConc mg/L	StdConc mg/L	Blncorr Signal	Time	Signal Stored
1	[4]	[4]	0.038	9:53:35 PM	Yes
2	[4]	[4]	0.042	9:53:39 PM	Yes
3	[4]	[4]	0.041	9:53:43 PM	Yes
Mean:	[4]	[4]	0.040		
SD:	0.00	0.00	0.0022		
%RSD:	0.00%	0.00%	5.33		

Standard number 3 applied. [4]
 Correlation Coef.: 0.995611 Slope: 0.01017 Intercept: 0.00000
 The calibration curve may not be linear.



Calibration data for Fe 248.33

Equation: Linear Through Zero

ID	Mean Signal (Abs)	Entered Conc. mg/L	Calculated Conc. mg/L	Standard Deviation	%RSD
Blank	0.000	0	0.000	0.00	0.35
Calib Std 1	0.012	1.0	1.191	0.00	41.13
Calib Std 2	0.020	2.0	1.941	0.00	19.61
Calib Std 3	0.040	4.0	3.972	0.00	5.33

Correlation Coef.: 0.995611 Slope: 0.01017 Intercept: 0.00000

Sequence No.: 20
Sample ID: 20
Analyst:

Autosampler Location:
Date Collected: 11/24/2011 9:54:24 PM
Data Type: Original

Replicate Data: 20 Sample ID: 0

Analyte: Fe 248.33

Repl #	Sample Conc mg/L	Std Conc mg/L	Blk Corr Signal	Time	Signal Stored
1	0.603	0.603	0.006	9:54:26 PM	Yes
2	1.243	1.243	0.013	9:54:30 PM	Yes
3	0.563	0.563	0.006	9:54:34 PM	Yes
Mean:	0.803	0.803	0.008		
SD:	0.3817	0.3817	0.0039		
%RSD:	47.53%	47.53%	47.53		

Sequence No.: 21
Sample ID: 1
Analyst:

Autosampler Location:
Date Collected: 11/24/2011 9:55:24 PM
Data Type: Original

Replicate Data: 1

Analyte: Fe 248.33

Repl #	Sample Conc mg/L	Std Conc mg/L	Blk Corr Signal	Time	Signal Stored
1	0.550	0.550	0.006	9:55:25 PM	Yes
2	0.271	0.271	0.003	9:55:29 PM	Yes
3	0.308	0.308	0.003	9:55:34 PM	Yes
Mean:	0.376	0.376	0.004		
SD:	0.1514	0.1514	0.0015		
%RSD:	40.25%	40.25%	40.25		

Sequence No.: 22
Sample ID: 5
Analyst:

Autosampler Location:
Date Collected: 11/24/2011 9:56:08 PM
Data Type: Original

Replicate Data: 5

Analyte: Fe 248.33

Repl #	Sample Conc mg/L	Std Conc mg/L	Blk Corr Signal	Time	Signal Stored
1	0.563	0.563	0.007	9:56:09 PM	Yes
2	0.282	0.282	0.002	9:56:13 PM	Yes
3	0.151	0.151	0.004	9:56:18 PM	Yes
Mean:	0.332	0.332	0.004		

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SD: 0.2653 0.2653 0.0027
 %RSD: 63.09% 63.09% 63.09

Sequence No.: 23
 Sample ID: 10
 Analyst:

Autosampler Location:
 Date Collected: 11/24/2011 9:56:55 PM
 Data Type: Original

Replicate Data: 10

Analyte: Fe 248.33

Repl #	SampleConc mg/L	StndConc mg/L	Blncorr Signal	Time	Signal Stored
1	0.046	0.046	0.000	9:56:57 PM	Yes
2	0.142	0.142	0.001	9:57:01 PM	Yes
3	0.535	0.535	0.005	9:57:05 PM	Yes
Mean:	0.241	0.241	0.002		
SD:	0.2588	0.2588	0.0026		
%RSD:	107.36%	107.36%	107.36		

Sequence No.: 24
 Sample ID: 15
 Analyst:

Autosampler Location:
 Date Collected: 11/24/2011 9:57:35 PM
 Data Type: Original

Replicate Data: 15

Analyte: Fe 248.33

Repl #	SampleConc mg/L	StndConc mg/L	Blncorr Signal	Time	Signal Stored
1	0.034	0.034	0.000	9:57:36 PM	Yes
2	0.733	0.733	0.007	9:57:40 PM	Yes
3	0.158	0.158	0.002	9:57:45 PM	Yes
Mean:	0.308	0.308	0.003		
SD:	0.3729	0.3729	0.0038		
%RSD:	120.92%	120.92%	120.92		

Sequence No.: 25
 Sample ID: 20
 Analyst:

Autosampler Location:
 Date Collected: 11/24/2011 9:58:14 PM
 Data Type: Original

Replicate Data: 20

Analyte: Fe 248.33

Repl #	SampleConc mg/L	StndConc mg/L	Blncorr Signal	Time	Signal Stored
1	0.690	0.690	0.007	9:58:16 PM	Yes
2	-0.058	-0.058	-0.001	9:58:20 PM	Yes
3	0.532	0.532	0.005	9:58:24 PM	Yes
Mean:	0.388	0.388	0.004		
SD:	0.3941	0.3941	0.0040		
%RSD:	101.51%	101.51%	101.51		

UMP

=====
Analysis Begun

Logged In Analyst: Administrator Technique: AA Flame
Spectrometer: AAnalyst 400, S/N 201S12010501 Autosampler:

Sample Information File: C:\Documents and Settings\All Users\PerkinElmer\AA\Data\Sample Information\
Cull1116Hong.sif

Batch ID:

Results Data Set: Cull1116Hong

Results Library: C:\Documents and Settings\All Users\PerkinElmer\AA\Data\Results\Results.mdb

=====
Method Loaded

Method Name: Cull1116Hong

Method Last Saved: 11/24/2011 9:23:44 PM

Method Description: Cull1116Hong

Sequence No.: 1

Autosampler Location:

Sample ID: Blank

Date Collected: 11/24/2011 9:28:17 PM

Analyst:

Data Type: Original

Replicate Data: Blank

Analyte: Cu 324.75

Repl #	SampleConc mg/L	StdConc mg/L	Blncorr Signal	Time	Signal Stored
1	[0.00]	0.137	0.137	9:28:19 PM	Yes
2	[0.00]	0.129	0.129	9:28:23 PM	Yes
3	[0.00]	0.132	0.132	9:28:28 PM	Yes
Mean:	[0.00]	0.133			
SD:	0.0000	0.0042			
%RSD:	0.00%	3.14			

Auto-zero performed.

Sequence No.: 2

Autosampler Location:

Sample ID: Calib Std 1

Date Collected: 11/24/2011 9:29:05 PM

Analyst:

Data Type: Original

Replicate Data: Calib Std 1

Analyte: Cu 324.75

Repl #	SampleConc mg/L	StdConc mg/L	Blncorr Signal	Time	Signal Stored
1	[1]	0.104	0.104	9:29:07 PM	Yes
2	[1]	0.105	0.105	9:29:11 PM	Yes
3	[1]	0.101	0.101	9:29:15 PM	Yes
Mean:	[1]	0.103			
SD:	0.00	0.0023			
%RSD:	0.00%	2.21			

Standard number 1 applied. [1]

Correlation Coef.: 1.000000 Slope: 0.10342 Intercept: 0.00000

Sequence No.: 3

Autosampler Location:

Sample ID: Calib Std 2

Date Collected: 11/24/2011 9:29:45 PM

Analyst:

Data Type: Original

Replicate Data: Calib Std 2

Analyte: Cu 324.75

Repl #	SampleConc mg/L	StdConc mg/L	Blncorr Signal	Time	Signal Stored
1	[2]	0.205	0.205	9:29:47 PM	Yes
2	[2]	0.203	0.203	9:29:51 PM	Yes
3	[2]	0.205	0.205	9:29:55 PM	Yes
Mean:	[2]	0.204			
SD:	0.00	0.0015			
%RSD:	0.00%	0.72			

Standard number 2 applied. [2]

Correlation Coef.: 0.999868 Slope: 0.10237 Intercept: 0.00000

=====

Sequence No.: 4
 Sample ID: Calib Std 3
 Analyst:

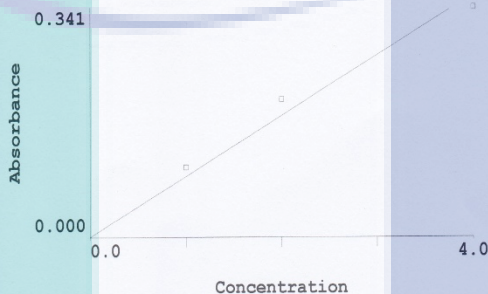
Autosampler Location:
 Date Collected: 11/24/2011 9:30:36 PM
 Data Type: Original

 Replicate Data: Calib Std 3

Analyte: Cu 324.75

Repl #	SampleConc mg/L	StndConc mg/L	BlkCorr Signal	Time	Signal Stored
1	[4]	0.344	0.344	9:30:37 PM	Yes
2	[4]	0.337	0.337	9:30:42 PM	Yes
3	[4]	0.342	0.342	9:30:46 PM	Yes
Mean:	[4]	0.341			
SD:		0.00	0.0038		
%RSD:		0.00%	1.12		

Standard number 3 applied. [4]
 Correlation Coef.: 0.984858 Slope: 0.08988 Intercept: 0.00000
 The calibration curve may not be linear.



 Calibration data for Cu 324.75

Equation: Linear Through Zero

ID	Mean Signal (Abs)	Entered Conc. mg/L	Calculated Conc. mg/L	Standard Deviation	%RSD
Blank	0.000	0	0.000	0.00	3.14
Calib Std 1	0.103	1.0	1.151	0.00	2.21
Calib Std 2	0.204	2.0	2.272	0.00	0.72
Calib Std 3	0.341	4.0	3.791	0.00	1.12

Correlation Coef.: 0.984858 Slope: 0.08988 Intercept: 0.00000

 Sequence No.: 5
 Sample ID: 0
 Analyst:

Autosampler Location:
 Date Collected: 11/24/2011 9:31:49 PM
 Data Type: Original

 Replicate Data: 0

Analyte: Cu 324.75

Repl #	SampleConc mg/L	StndConc mg/L	BlkCorr Signal	Time	Signal Stored
1	0.415	0.415	0.037	9:31:51 PM	Yes
2	0.390	0.390	0.035	9:31:55 PM	Yes
3	0.380	0.380	0.034	9:32:00 PM	Yes
Mean:	0.395	0.395	0.036		
SD:	0.0183	0.0183	0.0016		
%RSD:	4.63%	4.63%	4.63		

 Sequence No.: 6
 Sample ID: 1
 Analyst:

Autosampler Location:
 Date Collected: 11/24/2011 9:32:34 PM
 Data Type: Original

 Replicate Data: 1

Analyte: Cu 324.75

Repl #	SampleConc mg/L	StndConc mg/L	BlkCorr Signal	Time	Signal Stored
1	0.139	0.139	0.012	9:32:36 PM	Yes
2	0.143	0.143	0.013	9:32:40 PM	Yes
3	0.101	0.101	0.009	9:32:44 PM	Yes

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Mean: 0.128 0.128 0.011
 SD: 0.0230 0.0230 0.0021
 %RSD: 18.01% 18.01% 18.01

Sequence No.: 7

Sample ID: 5

Analyst:

Autosampler Location:

Date Collected: 11/24/2011 9:33:14 PM

Data Type: Original

Replicate Data: 5

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Analyte: Cu 324.75 Signal Stored
1	0.057	0.057	0.005	9:33:15 PM	Yes
2	0.038	0.038	0.003	9:33:19 PM	Yes
3	0.037	0.037	0.003	9:33:24 PM	Yes
Mean:	0.044	0.044	0.004		
SD:	0.0112	0.0112	0.0010		
%RSD:	25.50%	25.50%	25.50		

Sequence No.: 8

Sample ID: 10

Analyst:

Autosampler Location:

Date Collected: 11/24/2011 9:33:51 PM

Data Type: Original

Replicate Data: 10

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Analyte: Cu 324.75 Signal Stored
1	0.132	0.132	0.012	9:33:52 PM	Yes
2	0.102	0.102	0.009	9:33:56 PM	Yes
3	0.119	0.119	0.011	9:34:01 PM	Yes
Mean:	0.118	0.118	0.011		
SD:	0.0152	0.0152	0.0014		
%RSD:	12.90%	12.90%	12.90		

Sequence No.: 9

Sample ID: 15

Analyst:

Autosampler Location:

Date Collected: 11/24/2011 9:34:27 PM

Data Type: Original

Replicate Data: 15

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Analyte: Cu 324.75 Signal Stored
1	0.065	0.065	0.006	9:34:28 PM	Yes
2	0.137	0.137	0.012	9:34:32 PM	Yes
3	0.106	0.106	0.010	9:34:37 PM	Yes
Mean:	0.102	0.102	0.009		
SD:	0.0359	0.0359	0.0032		
%RSD:	35.03%	35.03%	35.03		

Sequence No.: 10

Sample ID: 20

Analyst:

Autosampler Location:

Date Collected: 11/24/2011 9:35:04 PM

Data Type: Original

Replicate Data: 20

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Analyte: Cu 324.75 Signal Stored
1	0.088	0.088	0.008	9:35:06 PM	Yes
2	0.137	0.137	0.012	9:35:10 PM	Yes
3	0.067	0.067	0.006	9:35:14 PM	Yes
Mean:	0.097	0.097	0.009		
SD:	0.0358	0.0358	0.0032		
%RSD:	36.88%	36.88%	36.88		