Studies of Zn(II) Ions Adsorption By Rice Husks Digested With Nitric Acid

By

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CERTIFICATION OF APPROVAL

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

Approved by,

(Normawati bt. Mohamad Yunus)

UNIVERSITI TEKNOLOGI PETRONAS TRONOH, PERAK January 2005

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CERTIFICATION OF ORIGINALITY

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

ang. DIANA SINGGAR

ABSTRACT

Rice husks are one of the agro residues which are available from mills in relatively dry form. It is a carbonaceous material and abundantly available especially in Malaysia. Therefore it is an innovative step to convert this abundantly renewable source into fully utilization. This particular research studies will demonstrate the adsorption capacity of Zn(II) ions in solution using activated carbon produced from rice husk. The adsorption capacity of activated carbon is not only determined by their physical or porous structure but also strongly influenced by the chemical structure of their surface. The usage of nitric acid is imposed to oxidize the rice husk hence extended its surface acidity. Surface acidity is categorized as the chemical structure that influences the carbon interaction with polar and non-polar adsorbates, which is, in this case is Zn(II) ion. Specifically, there are two main experiment to be conducted prior to the research studies which are; formation of activated carbon under the various experimental condition and the of Absorption analytical studies the adsorption capacity using Atomic Spectrophotometer (AAS). Carbons which have equal surface area but prepared by different methods or given different activation treatments show markedly different adsorption characteristics. This particular research project will demonstrate the effect of three variations of experimental conditions which are; weight ratio of Nitric acid to rice husks, shaker speed and contact time and finally carbonization time. From the experiment, it was found that the maximum yield of activated carbon was obtained from rice husks subjected to acid digestion of weight ratio of 2, carbonization period of 0.5 hours and final heat temperature of 500°C. As for the adsorption properties, the rice husks subjected to treatment at the weight ratio of 2, carbonization period of 3 hours and temperature of 500°C displayed the best adsorption tendency towards Zn(II) ions.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The purpose of these studies is to produce activated carbon from rice husks. The fundamental of activated carbon production is from the synthesis of organic matter that has high carbon content such as wood, coconut shell, bones, rice husks and coal. The synthesis process can be divided into two processes that are carbonization and activation¹.

The usage of activated carbon is widely applied in the industry where the treatment of wastewater is concerned. Activated carbon is used as the adsorbent through the adsorption process where the traces of metal such as Zn(II) ions that come into contact with it are trapped and hold on its internal surfaces. The degree of adsorption depends on the relationship between the carbon pore structures and the size and shape of the contaminating molecules¹.

1.2 Problem statement

In wastewater treatment process, the traces of metal are always removed using the mean of adsorption process. Malaysia is a developing country where its agriculture waste or byproduct always contribute to the environmental issues or left untended. Since the raw material for activated carbon is organic matter; therefore it is a wise step to fully utilize the agricultural waste and byproduct to form activated carbon hence to be applied in the wastewater treatment process⁴.

The formation of activated carbon from the agriculture residue is also economically feasible. This can be evaluated in term of its low production cost, renewable sources, and copious supplies and save to be used¹. Referring to the reasons stated, therefore the usage of activated carbon is obviously a wise choice.

For this research project, the activated carbon is produced from the digestion of rice husks with nitric acid in different experimental conditions.

1.3 Objectives

Prior to ensure that the research project can be completed and reasonable outcome are able to be achieved within the time scheduled, two main objectives have been identified.

The first objective is to study the effect of different preparation condition on the yields of the activated produced.

The second objective is to study the influence of different experimental condition on the adsorption capacity of the activated carbon produced. The different experimental condition is the contact time of the samples (zinc nitrate and activated carbons) and the shaker speed in which, of each of the samples is subjected to.

The final heat temperature treatment (HTT) of 500°C is selected for all experimental preparations and conditions.

1.4 Scope of Study

The scopes of work for the research project are stated as below:

- i. To conduct the experimental work for the formation of activated carbon from the rice husks.
- ii. To conduct the experimental observation on the Zn(II) adsorption capacity of the activated carbon produced at different experimental condition ie. weight ratio of nitric acid to precursor weight of rice husks, carbonization time to yield the activated carbon.
- iii. To study any possible factors that affect the adsorption rate.

CHAPTER 2

THEORY & LITERATURE REVIEW

2.1 Theory

In the sense of environmental protection, the utilization of agriculture waste such as rice husks has awakened the interest to study the process of forming carbon adsorbents based on agricultural wastes. Theoretically all carbonaceous materials are suitable for the formation of activated carbon subjected to proper treatment condition.

In activated carbon formation, two main processes involved that are carbonization and activation. Figure 1 previews the flow chart of activated carbon formation from any raw material.¹



Figure 1: The formation of activated carbon

2.1.1 Raw material

Activated carbon can be produced from any carbonaceous material. However, the preference is diverted to the material that posses high carbon content and low inorganic content. Referring to studies done by earlier scholars, the preferences in activated carbon is focused on younger fossil materials such as wood, peat, and washes of vegetables origin. The chars obtained from these materials can be used as the material to produce high-quality activated carbon. Current trend of activated carbon production is focused on two different factors namely on various type of materials which are cheap and readily available and also the utilization of carbonaceous wastes. The following criteria are considered when choosing a carbonaceous raw material.¹

- i. Potential of obtaining high-quality activated carbon
- ii. Presence of minimum inorganics
- iii. Volume and cost of the raw material
- iv. Storage life of the raw material
- v. Workability of the raw material.

2.1.2 Carbonization

Carbonization is defined as the decomposition of carbonaceous materials. Carbonization eliminates the noncarbon species and producing carbon at the fixed mass with rudimentary pore structures. The residual carbon atoms group themselves into sheets of condensed aromatic ring systems with a certain degree of planar structure. The mutual arrangement of these aromatic sheets is irregular and therefore leaves small voids or interstices between them that can be filled by the products of decomposition or at least blocked partially by the disorganized carbon. These small voids or interstices yield rise to pore that make activated carbons excellent adsorbents.¹

Carbonization is normally carried out as the temperature lower than 800°C in a continuous stream of an inert gas. This process created the effect of enlarging the pores size of the carbon due to the char shrinkage.

The following are the important parameter that determine the quality and the yield of the carbonized product.¹

- i. Rate of heating (carbonization time)
- ii. Final heat treatment temperature
- iii. Soaking time at final temperature
- iv. Nature and physical state of the raw material

2.1.3 Activation

Activation is the process done to enhance the volume and enlarge the pore's diameter and involves two main phases. In the first phase, the disorganized carbons are removed and the aromatic sheets are exposed to the action of activating agents. This action leads to the development of the microporous structure. In the second phase, the activated carbons are subjected to the complete burning process. The objective of this process is to widen the existence pores or increasing the formation of the largesized pores by completely burning the walls between the adjacent pores.¹ As the result from this process, the transitional pores and microporosity of the produced carbon grew larger hence the volume of the micropores grew smaller.

The in-depth studies of activation process will not be covered in this research studies. However, the information and the studies from previous scholars can be found in Bansal $(1998)^1$.

The activation process is divided into two categories namely chemical activation and physical activation. Chemical activation is normally imposed to raw material from wood origin. The starting material is digested with an activating agent (concentrated chemical solution) over a period of time to decompose the organic compound. The digested sample is then subjected to extrude and pyrolysis process in furnace under the pressure and at operating temperatures above 430°C in the absence of oxygen. In practice, it is not possible to achieve a completely oxygen-free atmosphere. Because some oxygen is present in any pyrolysis system, a small amount of oxidation occurs. If volatile or semi-volatile materials are present in the sample, thermal de-sorption will also occur.

The pyrolyzed product is then cooled and washed to remove the activating agent. In the current industry, the activating agents that are widely in used are phosphoric acid, zinc chloride and sulfuric acid. Physical activation is defined as the process of developing an extended surface area and porous structure of the carbonized material. Physical activation process normally executed at the operating temperature of 800°C to 1100°C in the presence of inert air. The main purpose of the inert air is to act as the oxidizing agent.

The heating process is executed using the mean of combustible of coke oven or natural gas. This is done in such a way because it is most economical as both the required heat and activation agent are supplied simultaneously.

Fundamentally, activated carbons are categorized based on the activation mode. Therefore, the activated carbon produced by chemical activation is known as chemical carbon and on the other hand, the activated carbon formed through physical activation is named as physical carbon.

2.2 Literature Review

The literature review is established based on the research studies done by previous scholars on the identical topic of research. Their significant findings and conclusion are used to elaborate the findings and justification on this respective research study.

Referring to the work of M.A Diaz et.al.² entitled, "Porous texture of activated carbons prepared by phosphoric acid activation of woods", two different concentrations of Phosphoric acid (H₃PO₄) were used as the activating agent. The concentrations are 36 wt.% H₃PO₄ and 85 wt.% of H₃PO₄. The activated carbons produced from three different raw materials are chestnut tree (Ch-36), cedar (Ce-36) and walnut (Wa-36) each are 50g of precursor from each of the samples were added to a 200 ml solution of H₃PO₄ and stirred at 85°C. The time for acid digestion was 4 hours. The same procedure were repeated for 85 wt.% of H₃PO₄. The impregnated materials were subjected to carbonization temperature of 450°C and carbonization time of 4 hours. The result obtained showed that the micropores volume (V_i) and specific surface area (SBET) values are quite similar in the H₃PO₄ 36 wt.%. series but lower and quite different in the 85wt.% of H₃PO₄ sample series. It was stated that solids in samples treated with 36wt.% of H₃PO₄ show an acceptable of microporosity and mesoporosity. The acid incorporated to the lignocellulosic structure of woods seems to be the cause of the creation of micropores. Thus, it can be concluded that the introduction of acid within the materials plays a double role to increase the material porosity. The first role, it produces hydrolysis of the lignocellulosic material and this weakens the particle, which swells. Secondly, the acid occupies a volume, which inhibits the contraction of the particle during the heat treatment, thus leaving porosity when it is extracted by washing after carbonization. The values of S_{BET} is reported to be lower at the temperature of 800°C.

In the research done by Castro et.al³ entitled, "Phosphoric acid activation of agricultural residues and bagasse from sugar cane: Influence of the experimental conditions on adsorption characteristics of activated carbon", the observation on the effect of three experimental conditions were made. The three experimental condition that were studied were the carbonization temperature (300°C - 600°C), the weight ratio of phosphoric acid to precursor (R = 1-2.5) and carbonization time (1-3 hours).

From the same studies, it was determined that the surface properties of the carbon formed were markedly dependent on the precursor and a combined effect of the conditions employed. The observation on the effect of heat temperature treatment (HTT) on the surface properties of the activated carbons showed two distinctive results where both surface area and pore volume are highly dependent on temperature. The activated carbons formed showed the maximum value of specific surface area (S_{BET}) and total pore volume at HTT of 500°C. Both properties have been identified to show the increasing trend towards attainment of a maximum value at certain value of HTT before decreasing with further temperature increment. It was observed at higher temperature, the microporous volume decreases and the macroporous volume increases. This observation can be explained using the properties of phosphoric acid properties. Phosphoric acid is imposed to induce chemical and structural alterations of the different constituent biopolymers of the precursors, namely cellulose, hemicellulose and lignin leading to the development of extensive porous structure. It was also reported that the phosphate ester cross-links reach their thermal stability temperatures at the temperature range of 450°C - 500°C. It was noted by the author that at higher temperatures, the break-down of the crosslink causing contraction and reducing in porosity development encountered. This particular observation is in parallel with the observation reported in M.A Diaz et.al.² , which stated the same observation at temperature greater than 450°C.

Based on the studies of Castro et.al³ on the effect of weight ratio on the surface properties of the activated carbons; the results showed higher surface area and total pore volumes were obtained at the weight ratio of 2 and weight ratio of 1.5 respectively. On top of these observations, it can be concluded that acid digestion promotes bond cleavage reactions and formation of new bio-phosphate ester links within the precursor structure which leads to a rigid matrix that restricts the shrinkage of the precursor and limits volatile matter lose upon heating. The insertion of phosphate groups brings about the dilation that leaves the cross-linked matrix in expanded state with an assessable porous structure. Higher impregnation ratios at each temperature led to reduction in sample porosity.⁴ However, it was noted that the impregnation ratio has its optimum value. The excess amount of acid used resulted in decrement of pore volumes.

Referring again to the research of Castro et.al³, the effect of carbonization time at 1 hour has resulted in maximum value of S_{BET} and total pore volumes. Based on facts stated by Castro et.al³, the carbon structures failed to achieve its maximum development at the carbonization less than 1 hour. The act of prolongation of the carbonization hours more than 1 hour has resulted in decrement of porosity. It was explained that the decrement of porosity is due to the breakdown of the cross-link within the carbon structures. It was suggested that less carbonization hours resulted in equally less energy and power consumption.

CHAPTER 3

METHODOLOGY

3.1 Experimental Procedure for the Preparation of Activated Carbon

- i. Obtain the rice husks sample from a rice mill.
- Dry, mill and screen-sieve the samples of rice husks. Fraction of particles in diameter range of 1mm was selected for preparation of activated carbon.
- Weigh 10g of the dried and sieved rice husks into a beaker. This is the dry weight of the rice husks.
- iv. Pour a certain volume of HNO₃ solutions into the beaker, resulting in weight ratios R, of HNO₃ acid to precursor of 1.
- v. Soak the rice husks in the acid for 20 hours at 25° C.
- vi. Wash the soaked rice husks using distilled water until the pH of the wash water is 5-6.
- vii. Dry the washed rice husks in an electrically heated oven (CARBOLITE) at 100° C to remove the free bound water.
- viii. Weigh the rice husks to obtain the weight of rice husks after digestion with the nitric acid.
 - ix. Transfer the rice husks into a crucible and insert the crucible into a hot furnace.
 - x. Heat the sample room temperature to the final heat treatment temperature (HTT) of 500°C.
- xi. Carbonize the impregnated samples in the air-enclosed with the sample in the furnace upon insertion, oxygen self-generated atmosphere, at HTT of 500°C.
- xii. Once desired HTT is attained, hold the final temperature for 0.5 hour.
- xiii. Cool the carbonized rice husks to room temperature in a dessicator.
- xiv. Weigh the crucible again to obtain the weight of rice husks after carbonization.

- xv. A series of activated carbon was prepared at selected weight ratio R (1-3) and varying carbonization time (0.5 3 hr).
- xvi. Store the prepared activated carbons in desiccators to minimize contact of adsorbent with ambient moisture.

3.2 Adsorption Studies of the Activated Carbon Produced

- i. Prepare a zinc nitrate, Zn(NO₃)₂ solution with Zn(II) ion concentration of 5 parts per million (ppm).
- 0.5 g of activated carbon produced under various conditions is mixed with 25ml of the zinc nitrate, Zn(NO₃)₂ solution in different beakers and subjected to different contact time and shaker speed as stated.
- iii. Shake the samples according to the designated time and speed by a shaker machine (GFL Model 3017).
- iv. Filter the samples and analyze the filtrates using the Atomic Absorption Spectrophotometer, (AAS).
- Determine the amount of Zn(II) ions adsorbed per gram of the activated carbon prepared by taking the difference between the initial known amount of Zn(II) ions in the solution and the residual amount of Zn(II) ions in the filtrate.

3.3 Tools and Chemicals Used

No.	Tool/Equipment
1.	Oven (CARBOLITE)
2	Shaker machine
3.	pH meter
4.	Atomic Absorption Spectrophotometer (AAS)

Table 1: Tool/Equipment required for the studies

No.	Chemicals/Material
1.	Nitric acid (65% provided by SYSTERM)
2.	Rice husk
3.	Zinc(II) Nitrate (provided by SYSTERM)

Table 2: Chemical/Material used

3.4 Atomic Absorption Spectrophotometer (AAS)

AAS is the main equipment used in these particular studies; therefore it is a good approach to give a brief introduction of the equipment and its function. The type of AAS used for these studies is Perkin-Elmer; hence this section will preview the general information of Perkin-Elmer Atomic Absorption Spectrophotometer.

Atomic absorption spectrophotometry works by providing the accurate quantitative analyses for metals in water, sediments, soils or rock. The samples are analyzed in the form of solution. Hence prior to analysis, the samples will need to be leached or dissolved⁷.

Atomics absorption units have four fundamental parts: interchangeable lamps that emit light with element - specific wavelengths, a sample aspirators, a flame or furnace apparatus for volatilizing the samples, and a photon detector⁷.



Figure 2: Schematic diagram of Atomic Absorption Spectrophotometer.

Prior to analyze any given element, a lamp that produces a wavelength of light that is absorbed by the element is selected. Sample solutions are aspirated into the flame and if any ions of the given element are present in the flame, they will absorb light produced by the lamp before it reaches the detector. The amount of the light absorbed depends on the amount of the element present in the sample. The absorbance values for the unknown samples are compared to the calibration curves or reference curves prepared by running known samples⁷.

CHAPTER 4

RESULT AND DISCUSSION

This section will discuss the result and finding on the first Experiment: Preparation of Activated Carbon from Rice husks and the second Experiment: Adsorption Studies of the Activated Carbon Produced.

4.1 Preparation of the Activated carbon from Rice Husks

Under this main title, two subsections will be discussed. The first subsection is the result of the activated carbon's formation and the second subsection is the discussion on the result obtained from the activated carbon's formation process.

4.1.1 Results from the Preparation of the Activated carbon from Rice Husks

The activated carbon was prepared according to the procedure listed. Two variation of experimental condition has been imposed namely the weight ratio of nitric acid to rice husks and carbonization time. Table 3 summarizes the activated carbons produced under the experimental parameters.

Table 3: Combination of parameter (Weight ratio & Carbonization time) under Final Heat Treatment of 500°C

Carbonization time	W	eight ratio	of Nitrie Aci	id to Rice h	usks (g/g)
	1	.0	2.	0	3	.0
0.5	A1	A2	B1	B2	C1	C2
1.0	A3	A4	B3	B4	C3	C4
3.0	A5	A6	B5	B6	C5	C6

Prior to study the effect of shaker speed and contact at the later part of this study, the activated carbon obtained in each of the combination parameter (weight ratio and carbonization time) is divided into two portion that each has the same amount. For example, activated carbon obtained at the combination parameter of weight ratio equals to 1.0 and carbonization hour of 0.5 hour is divided into samples labeled A1 and A2. Both samples are the same in quantity and composition. The same procedure is repeated for rest of the activated carbons produced under owns combination parameter.

Initially, three samples of rice husk weighted 10 g each were labeled A, B and C. Sample A, B and C were prepared under the stated experimental procedure and conditions (refer to Methodology) to yield the activated carbon.

Sample A, B and C then were divided into 6 samples and each sample will be carbonized according to the procedure listed, hence subjected to the adsorption studies of Zn(II) ions. The result of the studies is tabulated as follow.

 Table 4 : Summary of experimental data to calculate percent weight loss after acid

 digestion

	Weight ratio	Initial weight of dry rice husk (g)	Weight after free bound drying (g)	Weight loss after acid digestion (%)
A	1.0	10.0	8.4838	15.1620
В	2.0	10.0	8.3430	16.5700
С	3.0	10.0	8.0844	19.1560

Due to the scarcity of the rice husk, the additional samples were prepared. The remaining raw rice husks were grinded and sieved to produce rice husk of the size of 1mm. The same experimental procedure was repeated again.

	Weight ratio	Initial weight of dry rice husk (g)	Weight after free bound drying (g)	Weight loss after acid digestion (%)
Α	1.0	6.5987	5.2858	19.8963
B	2.0	6.5987	4.8426	26.6128
С	3.0	6.5987	4.6244	29.9195

Table 5: Summary of additional rice husks preparation

After the 20 hours of digestion using Nitric acid, the color or the rice husk changed from light yellow brownish to golden brownish. Very significant color changes were observed for sample digested with weight ratio of 3.

The first trial of activated carbon forming at final heat treatment temperature (HTT) of 500°C was failed. The sample was completely burnt into ashes. Based on the audit done on experimental condition during carbonization process, the crucibles were not fully covered with lids. Thus, due to the ingression of the atmosphere of the furnace, the rice husks were burnt instead of pyrolized. Theoretically, carbonization process should be carried out in a carbonization reactor where the inert stream of air is supplied continuously to ensure the proper carbonization of the sample. However, due to its unavailability, the hot furnace is utilized for the carbonization process.

After the first failure, extra precautions while carbonizing the sample were taken. The crucibles containing the digested sample were covered with lids. This is to minimize the ingression of the furnace atmosphere. As the result of this approach, the carbonized rice husks (activated carbon) were obtained.



Figure 3: Dried rice husks with Weight ratio 1.0



Figure 4: Dried rice husks with Weight ratio 2.0



Figure 5: Dried rice husks with Weight ratio 3.0



Figure 6: Activated carbon produced from Rice Husks

As mentioned earlier, due to the scarcity of the rice husk, the 18 samples were produced by carbonizing 2g of rice husk to yield approximately 0.3g to 0.6g of activated carbon. The experimental results obtained are tabulated as follow.

Sample	Weight	Carbonization	Initial	Weight of	Activated
	Ratio(g	time (nr)	weight of	rice husks	carbon yield
	NHFIC		aried rice	atter	(%)
	Acid/g rice		husks (g)	carbonizati	
	husks)	· · · · · · · · · · · · · · · · · · ·		on(g)	
<u>A1</u>	1.0	0.5	2.0	0.6906	34.53
B 1	2.0	0.5	2.0	0.7436	37.18
C1	3.0	0.5	2.0	0.7258	36.29
A2	1.0	0.5	2.0	0.7040	35.20
B2	2.0	0.5	2.0	0.7502	37.51
C2	3.0	0.5	2.0	0.7260	36.30
A3	1.0	1.0	2.0	0.6682	33.41
B3	2.0	1.0	2.0	0.6590	32.95
C3	3.0	1.0	2.0	0.7264	36.32
A4	1.0	1.0	2.0	0.6654	33.27
B 4	2.0	1.0	2.0	0.5495	27.48
C4	3.0	1.0	2.0	0.7148	35.74
A5	1.0	3.0	2.0	0.3632	18.16
B5	2.0	3.0	2.0	0.3578	17.89
C5	3.0	3.0	2.0	0.3416	17.08
A6	1.0	3.0	2.0	0.2866	14.30
B 6	2.0	3.0	2.0	0.2766	13.88
C6	3.0	3.0	2.0	0.3297	16.49

Table 6: Summary of experimental data to calculate activated carbon yield after acid digestion and carbonization

Sample	Weight Ratio(g Nitric Acid/g rice husks)	Carbonization time (hr)	Initial weight of dried rice husks (g)	Weight of rice husks after carbonizati on(g)	Percent weight loss after acid digestion (%)
<u>A1</u>	1.0	0.5	2.0	0.6906	65.47
B1	2.0	0.5	2.0	0.7436	62.82
<u>C1</u>	3.0	0.5	2.0	0.7258	63.71
<u>A2</u>	1.0	0.5	2.0	0.7040	64.80
<u>B2</u>	2.0	0.5	2.0	0.7502	62.49
<u>C2</u>	3.0	0.5	2.0	0.7260	63.70
<u>A3</u>	1.0	1.0	2.0	0.6682	66.59
<u>B3</u>	2.0	1.0	2.0	0.6590	67.05
<u>C3</u>	3.0	1.0	2,0	0.7264	63.68
<u>A4</u>	1.0	1.0	2.0	0.6654	66.73
<u> </u>	2.0	1.0	2.0	0.5495	72.53
<u>C4</u>	3.0	1.0	2.0	0.7148	64.26
<u>A5</u>	1.0	3.0	2.0	0.3632	81.84
<u>B5</u>	2.0	3.0	2.0	0.3578	82.11
<u>C5</u>	3.0	3.0	2.0	0.3416	82.92
<u>A6</u>	1.0	3.0	2.0	0.2866	85.67
B6	2.0	3.0	2.0	0.2766	86.17
C 6	3.0	3.0	2.0	0.3297	83.52

Table 7: Summary of experimental data to calculate percent weight loss after acid digestion



Figure 7: Graph of Weight Loss (%) vs. Weight Ratio of Nitric Acid to Rice Husks (g/g) After Acid digestion



Figure 8: Graph of Activated Carbon Yield (%) vs. Carbonization Time (hr)

4.1.2 Discussion on the result of activated carbon formation from rice husks.

The rice husks samples were divided into 6 groups. Each of the group consists of 3 samples each. These samples are subjected to the digestion with Nitric acid of 3M at the weight ratio of 1, 2 and 3. Prior for carbonization process, Group 1 and Group 2 were subjected to the carbonization time of 0.5 hour, Group 3 and Group 4 were subjected to carbonization time of 1 hour and Group 5 and Group 6 were subjected to carbonization time of 3 hours. It was hypothesized that the weight loss is proportional to the weight ratio.

By Referring to Figure 7; Group 5 shows the most distinctive pattern compare to others and regard as the best result. The pattern of increment in weight loss proportional to increment in weight ratio is clearly seen. Referring to Group 5, which is labeled as Series 5 (A5, B5 and C5) in the respective graph; the trend of increment in weight loss as the weight ratio increases can be clearly noted. By closer observation, sample A5 which was subjected to digestion with Nitric acid at weight ratio of 1 established lesser amount of weight loss as compare to sample C5 which was subjected to acid digestion at the weight ratio of 3. Again, it supported the hypothesis made.

However, the discrepancy of pattern also noted. Sample B6 which was subjected to digestion with Nitric acid of weight ratio of 2 exhibit the % weight loss higher than sample C6. The same observation also noted in Series 1 and Series 2, where sample A1 illustrated an amount of weight loss at 65.47 % compare to sample C1 which previewing the weight loss of 63.71 %. These inaccurate data obtained are presumably due to the loss of rice husk during the washing with distilled water prior to its drying process in the oven.

Overview of the result obtained; the hypothesis made is in parallel with the trend obtained in the graph.

Weight Ratio(g Nitric Acid/g rice husks)	Average Weight Loss After Digestion
1.0	17.53
2.0	21.59
3.0	24,54

Table 8: Summary of the Average Weight Loss of Rice Husks after Acid Digestion

Table 8 illustrates the ascending trend of average weight loss to the weight ratio of Nitric acid to rice husks.

This particular observation can be explained by the previous graph. Referring to the research studies conducted by Mudjijati et.al⁵; rice husks are mainly made of organic materials such as cellulose, hemicellulose and also lignin which are easily digested by nitric acid. Based on the research studies made by the author, it was concluded that nitric acid are able to digest the materials over period of time. Therefore it is justified that the weight losses in rice husks are contributed by this factor.

4.2 Adsorption Capacity Studies of the Prepared Activated Carbon

Under this main title, the result of the adsorption test of Zn(II) ions from $Zn(NO_3)_2$ solution will be further discussed. The result obtained will be analyzed with reference to the previous studies done by scholars on similar topic to justify the findings.

4.2.1 Results from the Adsorption Test

As indicated in the problem statement earlier, the main focus of this research study is to remove the heavy metals in wastewater at the trace level. Referring to the work of I.A Rahman et al.⁶, the adsorption process is imposed to remove the heavy metal in the wastewater at the trace level. Based on this objective, various materials have

been identified as the possible raw material for the formation of activated carbon such as saw dust, coconut shell and rice husk. Zn (II) ions have been selected as the adsorbate for the adsorption process because the ions are commonly found to be available in the wastewater at the trace level.

Hence, to adhere the idea of I.A Rahman et. al^6 , the usage of Zn(II) ions also imposed as the adsorbate in this project. The solution of $Zn(NO_3)_2$ that has the concentration of 5 part per million (ppm) is prepared in a way that the concentration of 5ppm is the trace level that is commonly found in wastewater.

The activated carbons formed under various experimental condition were subjected to the adsorption process to study its adsorption capacity towards Zn(II) ions. The summary of the adsorption capacities of the activated carbons is tabulated in Table 9.

Sample	Contact time (min)	Shaker speed (rpm)	Adsorption capacity (mg Zn(II) ions adsorbed/g activated carbon)
A1	30	100	0.0164
A2	60	200	0.0278
A3	30	100	0.0145
A4	60	200	0.0129
A5	30	100	0.0415
A6	60	200	0.0070
B1	30	100	0.0234
B2	60	200	0.0225
B3	30	100	0.0130
B 4	60	200	0.0528
B5	30	100	0.0537
B6	60	200	0.0295
C1	30	100	0.0482
C2	60	200	0.0165
C3	30	100	0.0205
C4	60	200	0.0355
C5	30	100	0.0290
C6	60	200	0.0450

Table 9: Summary of Adsorption Capacities of the Activated Carbons



Figure 9: Graph of Adsorption Capacity vs. Carbonization Time at Shaker speed 100rpm and contact time 0.5 hr



Figure 10: Graph of Adsorption Capacity vs. Carbonization Time at Shaker speed 200rpm and contact time 1.0 hr

4.2.2 Discussion on the Adsorption Test Results

The discussion is based on Figure 9 because it previews the theoretical trend of adsorption capacity towards metal ions.

The various samples namely Series 1(A1, A3 and A5), Series 2(B1, B3 and B5) and Series 3 (C1, C3 and C5). Each of the series is subjected to the specific weight ratio of Nitric acid to rice husks. As each of the series are subjected to the carbonization time that varies from 0.5 hour, 1 hour and 3 hour. It can be observed that the adsorption capacity decreases at the carbonization time of 0.5 hour to carbonization time of 1 hour and later showed a rapid increment as it is subjected to longer carbonization period of 3 hours. Therefore, the first importance point to be noted is that the adsorption capacity increases as the carbonization period increases.

The second point to be noted from the same figure is that at a particular carbonization period, as the weight ratio of nitric acid to rice husk increases, the corresponding increment in the adsorption capacity for each of the series is observed. The trend is clearly illustrated at the carbonization period of 1 hour to the carbonization period of 3 hours. However, the similar trend is not observed at the carbonization period lower than 1 hour.

Thus, the two distinctive points observed in Figure 9 can be explained with reference to the work of previous scholars on the identical research studies.

Referring to the work of Castro et.al³, it was reported that at shorter carbonization period, the carbon structures are still very much intact and it has not achieved the full development in porosity. Hence, to support the comment stated by the author, increasing the contact time and enhance the surface contact between the carbons and adsorbate material through shaking the mixture; did not help much prior to obtain higher adsorption capacity. Thus, it can be concluded, at shorter carbonization period less than 1 hour, the highly microporous activated carbons were obtained. Hence, the trend observed in Figure 9 is explained.

Again, referring to the work of Castro et.al³, it was stated that as the carbonization period increases, the same observation were obtained for the formation of mesoporous volume. However, the increment of the mesoporous volume also decreasing once the optimum value is attainable. As the carbonization period increases, the notable increment in the adsorption capacity of all of the series. Hence, this observation justifying that the adsorptions toward Zn(II) ions are more favourable at higher mesoporous volume. Therefore, the trend observed is Figure 9 once again justified.

Judging the condition of decreasing trend to the work of Castro et.al.³, it is presumed to be the effect of carbon's cross-link breakdown. Indicated in the studies of Castro et.al³, prolongation of the carbonization period exceeding the time of 1 hour leads to the decrement in porosity. The decrement in porosity development is signified by the decrement in surface areas and total pore volume of the activated carbons. This particular observation is sternly emphasized by the trend of Series 1 and Series 2 else, Series 3 falls into the category of trend discussed based on Figure 9.

Taking Figure 10 into consideration, the mix trends of decreasing and increasing of the adsorption capacity for Series 1, Series 2 and Series 3 past the carbonization period of 1 hour are observed. Hence, to avoid the misconception of the possible factors that affected the adsorption rate, therefore further studies is recommended to be executed.

Sample	Preparation	Conditions	Adsorption Capacity,(mg
*	Carbonization time (hr)	Weight ratio of HNO3 to rice husks(g/g)	Zn(II) ions adsorbed/g activated carbon)
A1	0.5	1.0	0.0164
B1	0.5	2.0	0.0234
C 1	0.5	3.0	0.0482
A2	0.5	1.0	0.0278
B2	0.5	2.0	0.0225
C2	0.5	3.0	0.0165
A3	1.0	1.0	0.0145
B 3	1,0	2.0	0.0130
C3	1.0	3.0	0.0205
A4	1.0	1.0	0.0129
B4	1.0	2.0	0.0528
C4	1.0	3.0	0.0355
A5	3.0	1.0	0.0415
B 5	3.0	2.0	0.0537
C5	3.0	3.0	0.0290
A6	3.0	1.0	0.0070
B6	3.0	2.0	0.0295
C 6	3.0	3.0	0.0450

Table 10: Summary of Adsorption Capacities of All Activated Carbon Prepared under various experimental Conditions

Referring to Table 10, the highest adsorption capacity is 0.0537 mg Zn(II) ions adsorbed/g of activated carbon. The shaded box indicates the highest adsorption capacity among the 18 samples prepared. The sample that possesses the highest adsorption capacity is sample labeled B5, produced under the digestion of Nitric acid of weight ratio 3 and subjected to carbonization time of 3 hours. The second highest adsorption capacity was obtained from sample B4 with value of 0.0528 mg Zn(II) ions adsorbed/g of activated carbon. Judging the two samples can gave highest amount of Zn(II) ions adsorbed, it can be seen clearly that samples were produced at the weight ratio of nitric acid to rice husks of 2. Thus, it can be concluded and recommended that preparation method at weight ratio of 2 is favorable because it requires less chemical hence cost effective.

From Table 10 also, it was noted that there are inconsistency of adsorption capacity at as it subjected to longer carbonization time. Taking the example of samples subjected to the carbonization time of 3 hours; the sample digested at the weight ratio of 1 showed higher adsorption capacity than the sample digested at the weight ratio of 3. This particular observation however deviated from the hypothesis that the adsorption capacity increases as the carbonization time increases. In sequential to the work done by Castro et.al.³, the particular observation can be justified. It was reported in his studies that the beneficial effect of increasing the impregnation ratio(it this case, weigh ratio of nitric acid to rice husks) reaches the optimum point at which the excess of acid resulted in collapse of pores hence induce structural weakness.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

The maximum yield of activated carbon was obtained from rice husks that were subjected to carbonization period of 0.5 hours and acid digestion of weight ratio 2. The amount of activated carbon yield was 37.51%.

The highest adsorption capacity was obtained from rice husks that were subjected to acid treatment at weight ratio of 2 with carbonization period of 3 hours, shaker speed of 100 rpm and contact time of 0.5 hours.

From the research studies conducted, it can be concluded that, the adsorption capacity is proportional to the weight ratio of nitric acid to rice husks. However, this increment has an optimum value. If the optimum value is exceeded, the adsorption capacity of the activated carbons will decrease.

Based on the studies conducted, it was proven that rice husks have the potential as the raw material for activated carbon formation, hence referring to the fact few recommendations are made to further improve the findings on this current work.

First, it is recommended that various ratio of activating agent to be identified. It is suggested in a manner that the effect of using both low and high weight ratio of activating agent to rice husk can be analyzed hence the optimum value of ratio can be identified.

Second, it is recommended that various type of chemical activating acid to be identified. It is suggested in such a way that, high quality of activated carbon can be produced at the low volume of activating agent used.

Third, it is recommended that the range carbonization time to be extended or prolonged. It is suggested with respect to the earlier studies done by scholars who cited that the adsorption capacity increases as the carbonization time increases. This particular approach also enables the investigation on the declining point of adsorption capacity towards thermal degradation of the carbon structure to be determined.

Fourth, it is recommended to study the surface properties of each samples produced. The surface properties such as pore volume and surface area surface need to be determined hence the in-depth knowledge on the effect of carbonization time, weight ratio, and adsorption capacity and also the effect of contact time can be sought. The additional advantage is that the current understanding on activated carbon formation can be improved.

Fifth, is recommended to use the incorporation of advanced technology to enhance the investigation of the activated carbon properties such as Boehm method. Boehm method is used to assess the chemical changes mainly to determine the surfaceacidic groups of the carbons.

Lastly, further studies on this particular topic are recommended to be executed to improve the steps in formulating good quality of activated carbon to be used in various industries.

REFERENCES

- 1. Roop Chand Bansal, Jean-Baptiste Donnet, Frix Stoeckli, 1988, Active Carbon, USA, Marceldekker Inc.
- M.A. Diaz-Diez, V. Gomez-Serrano, C. Fernandez Gonzalez, E.M Cuerda-Correa and A. Macias-Garcia, 2004, "Porous texture of activated carbons prepared by phosphoric acid activation of woods," *Applied Surface Science*, 238(2004):309-313
- Javier Blanco Castro, Pablo R. Bonelli, Elsa G. Cerella and Ana L. Cukierman, 2000, "Phosphoric Acid Activation of Agricultural Residues and Bagasse from Sugar Cane: Influence of Experimental Conditions on Adsorption Characteristics of Activated Carbons," *Industrial Engineering and Chemical Research*, 39:4166-4172.
- 4. Ng Joon Leong, January 2004, Nitric Acid Activation of Rice Husks: Influence of the experimental Conditions on the Adsorption Capacities of the Activated Carbons; University Technology of PETRONAS.
- Mudjijati, N.Indraswati, F.Wicaksana, Herman, I.Harsono and Ismadi, 2000, "Activated Carbon Produced from Kalimantan Peat by Chemical Activation." Adsorption Science and Technology, pg 446-450
- I.A Rahman, J. Ismail and H. Osman, 2000, "Studies of Zn(II) Ions Adsorption by Rice husks Digested with Nitric Acid," *Malaysian Journal of Chemistry*, Vol. 2 No. 1, 012-015
- 7. http://departments.colgate.edu/geology/instruments/aa.htm

APPENDIX I : Results from Activated Carbon formation

Sample	Weight Ratio(g Nitric Acid/g rice	Carbonization time (hr)	Initial weight of dried rice husks (g)	Weight of rice husks after carbonizati on(g)	Percent weight loss after acid digestion (%)
<u> </u>		0.5	2.0	0.6906	65.47
A1 D 1	2.0	0.5	2.0	0.7436	62.82
	3.0	0.5	2.0	0.7258	63.71
<u>42</u>	10	0.5	2.0	0.7040	64.80
B2	2.0	0.5	2.0	0.7502	62.49
<u>C2</u>	3.0	0.5	2.0	0.7260	63.70
A3	1.0	1.0	2.0	0.6682	66.59
B3	2.0	1.0	2.0	0.6590	67.05
C3	3.0	1.0	2.0	0.7264	63.68
A4	1.0	1.0	2.0	0.6654	66.73
B 4	2.0	1.0	2.0	0.5495	72.53
C4	3.0	1.0	2.0	0.7148	64.26
A5	1.0	3.0	2.0	0.3632	81.84
B 5	2.0	3.0	2.0	0.3578	82.11
C5	3.0	3.0	2.0	0.3416	82.92
A6	1.0	3.0	2.0	0.2866	85.67
B6	2.0	3.0	2.0	0.2766	86.17
C6	3.0	3.0	2.0	0.3297	83.52

Table A.I.1: Summary of experimental data to calculate percent weight loss after acid digestion

Referring to Table A.I1, The percentage of weight loss after acid digestion is calculated according to the following formula:

% Weight Loss = <u>Initial weight of dried rice husks - Weight of rice husks after digestion</u> X 100 Initial weight of dried rice husks

Sample	Weight Ratio(g Nitric Acid/g rice husks)	Carbonization time (hr)	Initial weight of dried rice husks (g)	Weight of rice husks after carbonizati on(g)	Activated carbon yield (%)
A1	1.0	0.5	2.0	0,6906	34.53
B1	2.0	0.5	2.0	0.7436	37.18
C1	3.0	0.5	2.0	0.7258	36.29
A2	1.0	0.5	2.0	0.7040	35.20
B2	2.0	0.5	2.0	0.7502	37.51
C2	3.0	0.5	2.0	0.7260	36.30
A3	1.0	1.0	2.0	0.6682	33.41
B 3	2.0	1.0	2.0	0.6590	32.95
C 3	3.0	1.0	2.0	0.7264	36.32
A4	1.0	1.0	2.0	0.6654	33.27
B4	2.0	1.0	2.0	0.5495	27.48
C4	3.0	1.0	2.0	0.7148	35.74
A5	1.0	3.0	2.0	0.3632	18.16
B5	2.0	3.0	2.0	0.3578	17.89
C5	3.0	3.0	2.0	0.3416	17.08
A6	1.0	3.0	2.0	0.2866	14.30
B6	2.0	3.0	2.0	0.2766	13.88
C6	3.0	3.0	2.0	0.3297	16.49

 Table A.I.2: Summary of experimental data to calculate activated carbon yield after acid

 digestion and carbonization.

Referring to Table A.I.2, the yield of the activated carbon prepared under various experimental conditions can be computed using the following formula.

% Activated carbon yield = $\{1 - \frac{\text{Initial weight of rice husks} - \text{Weight of rice husks after carbonization}}{\text{Initial weight of rice husks}}\} \times 100$

APPENDIX II: Results to Calculate Adsorption Capacities

The details are the detail obtained from the analysis conducted using Atomic Absorption Spectrophotometer. Inclusive are the calculations that led to the values as tabulated.

Sample	Carboni zation time (hr)	Weight Ratio(g Nitric Acid/g rice husks)	Sample Weights (g)	Residue concentrati on Of Zn(II) ion (mg/I)	Initial concentra tion Of Zn(II) ion (mg/I)	Amount of Zn(II) adsorbed (mg)	Adsorption Capacity,(mg Zn(II) ions adsorbed/g activated carbon)
A 1	0.5	10	0 5034	2.67	3.00	0.00825	0.0164
R1	0.5	2.0	0.5021	2.53	3.00	0.01175	0.0234
<u> </u>	0.5	3.0	0.5029	2.03	3.00	0.02425	0.0482
A2	0.5	1.0	0.5039	2.44	3.00	0.01400	0.0278
B2	0.5	2.0	0.5004	2.55	3.00	0.01125	0.0225
<u>C2</u>	0.5	3.0	0.5004	2.67	3.00	0.00825	0.0165
A3	1.0	1.0	0.5000	2.71	3.00	0.00725	0.0145
B3	1.0	2.0	0.5014	2.74	3.00	0.00650	0.0130
C3	1.0	3.0	0.5004	2.59	3.00	0.01025	0.0205
A4	1.0	1.0	0.5020	2.74	3.00	0.00650	0.0129
B 4	1.0	2.0	0.5495	1.84	3.00	0.02900	0.0528
C4	1.0	3.0	0.5003	2.29	3.00	0.01775	0.0355
A5	3.0	1.0	0.5000	2.17	3.00	0.02075	0.0415
B 5	3.0	2.0	0.4981	1.93	3.00	0.02675	0.0537
C5	3.0	3.0	0.5000	2.42	3.00	0.01450	0.0290
A6	3.0	1.0	0.5000	2.86	3.00	0.00350	0.0070
B6	2.0	3.0	0.5000	2.41	3.00	0.01475	0.0295
C6	3.0	3.0	0.5000	2.10	3.00	0.0225	0.0450

Table A.II.1: Summary of experimental data to calculate the adsorption capacities

Referring to Table A.II.1, the main interest is to find the adsorption capacity of each one of the activated carbon produced under various experimental conditions. The adsorption capacities were calculated using the formula as follow.

The residue concentration of Zn(II) ions is the remaining Zn(II) ions in the $Zn(NO_3)_2$ solution after the adsorption by activated carbon formed. The value of the residues was given by the AAS. The printouts displaying the residue concentration are appended in Appendix III.

The initial concentration of Zn(II) ions is the concentration of Zn(II) ions of the $Zn(NO_3)_2$ solution prepared. Theoretically, the initial concentration of 5.00ppm supposes to be prepared hence analysis done using AAS showed the concentration to be 3.00ppm.

This particular error however can be justified due to the type of chemical used. The chemical, $Zn(NO_3)_2$ in the form of solid were provided by SYSTERM. SYSTERM chemicals are also known as repacked chemical, which mean the chemicals are subjected to high chances of oxidation prior to the repacking process. Thus, the quality of chemical has been contaminated. Hence, this explained why the concentration of the solution prepared deviated from 5.00ppm to 3.00ppm.

A volume of 25 ml of zinc nitrate was used. Thus,

Amount of Zn(II) ions adsorbed = $\frac{25}{1000}$ [Initial Concentration – Residue Concentration]

Adsorption Capacity = $\frac{\text{Amount of } Zn(II) \text{ ion adsorbed}}{\text{Sample weight}}$

All the units in the equations used will be those as tabulated in Table A.II.1.

APPENDIX III: Results from the AAS

The following printout data are the analysis obtained from the Atomic Absorption Spectrophotometer. In each of the printouts, the data that need to be extracted is the mean concentration of Zn(II) ion in the solution analyzed. This concentration is identified as the residue concentration - concentration of the ion after adsorption by the activated carbon produced.

Detail Table

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Analysis M Analysis N Comment	ode : ame : :	Flame/Autosa Studies of 2 Diana Singga	ampler Zn(II) ion ads ar	orption by ri	ice husk	
Meas. Date :10/11/04 3:07 PM						
Sample ID No. 1 Mean SD	:STD1 Corr.	Conc.	Sample Name: Conc.(ppm) 0.00 	ABS -0.0001 -0.0001 0.0000	REF -0.0043 -0.0043 0.0000	
RSD(%)				0.00	0.00	
Sample ID No. 1 Mean SD RSD(%)	:STD2 Corr. 	2 .Conc.	Sample Name: Conc.(ppm) 0.20 	ABS 0.0497 0.0497 0.0000 0.00	REF 0.0112 0.0112 0.0000 0.00	
Sample ID No. 1 Mean SD RSD(%)	:STD: Corr 	3. Conc.	Sample Name: Conc.(ppm) 0.40 	ABS 0.1045 0.1045 0.0000 0.00	REF 0.0201 0.0201 0.0000 0.00	
Sample_LD No. 1 Mean SD RSD(%) Coefficien	:STD Corr nt :K	4 .Conc. 3=	Sample Name: Conc.(ppm) 0.80 	ABS 0.2305 0.0000 0.000	REF 0.0374 0.0374 0.0000 0.000	
Corr.Coef	К К К . :0	2= 1=2.900857E- 0=-5.379999E .9985	.001 ;-003	···		
				•		
Sample ID No. 1 Mean SD RSD(>)	:UNK Corr 3.00 3.00 0.00 0.00	-001 .Conc.(ppm)))	Sample Name Conc.(ppm) 3.00 3.00 0.00 0.00	standard sol: ABS 0.8646 0.8646 0.0000 0.000	Lutiaon 2 REF C 0.3944 0.3944 0.0000 0.00	
Sample ID No. 1 Mean SD RSD(%)	 UNK Corr 3.00 3.00 0.00 0.00 	(-002 c.Conc.(ppm))))	Sample Name Conc.(ppm) 3.00 3.00 0.00 0.00	standard sol: ABS 0.8653 0.8653 0.0000 0.00	lution 3 REF C 0.3929 C 0.3929 0.0000 0.000	

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Analysis Mode :Flame/Autosampler Analysis Name : Studies of Zn(II) ion adsorption by rice husk :Diana Singgar Comment Meas. Date :10/11/04 10:37 AM Element :Zn Sample ID :STD1 . Sample Name: No. Corr.Conc. Conc. (ppm), ABS REF 0.00 -0.0004-0.0055 1 __ : --___ -0.0004 -0.0055 Mean 0.0000 0.0000 SD _ _ _ <u>`</u> 0.00 0,00 RSD(%) ___ ----Sample ID :STD2 Sample Name: ABS REF No. Corr.Conc. Conc. (ppm) 0.0345 0.20 0.0050 1 _ _ 0.0345 0.0050 Mean _ _ _ _ 0.0000 0.0000 _ _ SD _ _ 0.00 0.00 ---RSD(\$) _ _ Sample ID :STD3 Sample Name: Conc. (ppm) ABS REF No. Corr.Conc. 1 ___ 0.40 0.0972 0.0161 Mean --------0.0972 0.0161 0,0000 0.0000 SD -----_ _ 0.00 0.00 RSD(S)_ _ ___ Sample ID :STD4 Sample Name: 1 ABS REF No. Corr.Conc. Conc. (ppm) 0.1833 0.80 0.0271 1 _ _ 0.1833 0.0271 _ _ _ ----Mean 0.0000 0.0000 ---------SD 0.00 0.00 ----_ _ RSD(%) Coefficient :K3=--. K2=--• K1=2.351714E-001 K0=-3.660001E-003 :0.9965 Corr.Coef. inter: Sample Name:standard solution Sample ID :UNK-001 ABS REF Conc. Xppn No. Corr.Conc. (ppm) 1.36 0.3152 C 1.2311 1 1.36 1.36 0.3152 1.2311 1.36 Mean 0.00 0.0000 0.0000 0.00 SD 0.00 0.00 0.00 RSD(S)0.00 Sample Name:Al Sample ID :UNK-002 Conc. (ppm) ABS REF Corr.Conc. (ppm) No. 0.6250 C 0.8075 2.67 1 2.67 2.67 0.6250 0.8075 2.67 Mean 0.0000 0.0000 0.00 0.00 SD 0.00 0.00 0.00 RSD(1) 0.00 Sample ID :UNK-003 Sample Name:A2 REF ARS Conc. (ppm) Corr.Conc. (ppm) No. 0.5698 C 0.8850 2.44 2.44 1 0.5698 0.8850 2.44 2.44 Mean 0.0000 0.0000 0.00 0.00 SD 0.00 0.00 0.00 RSD(3) 0.00 Sample Na:42:A3 Sample ID :UNK-004 ABS REF Corr.Conc. (ppm) Conc. (ppm) No. 0.6343 C 0.7860 2.71 1 2.71

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Mean SD RSD(%)	2.71 0.00 0.00	2.71 0.00 0.00	0.6343 0.7860 0.0000 0.0000 0.00 0.00
Sample ID No. 1 Mean SD RSD(%)	:UNK-005 Corr.Conc.(ppm) 2.74 2.74 0.00 0.00	Sample Name:A4 Conc.(ppm) 2.74 2.74 0.00 0.00	ABS REF 0.6399 C 0.7870 0.6399 0.7870 0.0000 0.0000 0.00 0.00
Sample ID No. 1 Mean SD RSD(%)	:UNK-006 Corr.Conc.(ppm) 2.53 2.53 0.00 0.00	Sample Name:B1 Conc.(ppm) 2.53 2.53 0.00 0.00	ABS REF 0.5923 C 0.8500 0.5923 0.8500 0.0000 0.0000 0.00 0.000
Sample ID No. 1 Mean SD RSD(%)	:UNK-007 Corr.Conc.(ppm) 2.55 2.55 0.00 0.00	Sample Name:B2 Conc.(ppm) 2.55 2.55 0.00 0.00	ABS REF 0.5953 C 0.8505 0.5953 0.8505 0.0000 0.0000 0.00 0.00
Sample ID No. 1 Mean SD RSD(%)	:UNK-008 Corr.Conc.(ppm) 2.74 2.74 0.00 0.00	Sample Name:B3 Conc.(ppm) 2.74 .2.74 0.00 0.00	ABS REF 0.6400 C 0.7806 0.6400 0.7806 0.0000 0.0000 0.000 0.000
Sample ID No. 1 Mean SD RSD(%)	:UNK-009 Corr.Conc.(ppm) 2.03 2.03 0.00 0.00	Sample Name:C1 Conc.(ppm) 2.03 2.03 0.00 0.00	ABS REF 0.4733 C 1.0099 0.4733 1.0099 0.0000 0.0000 0.00 0.00
Sample ID No. 1 Mean SD RSD(7)	:UNK-010 Corr.Conc.(ppm) 2.67 2.67 0.00 0.00	Sample Name:C2 Conc.(ppm) 2.67 2.67 0.00 0.00	ABS REF 0.6244 C 0.8058 0.6244 0.8058 0.0000 0.0000 0.000 0.000 i
Sample ID No. 1 Mean SD RSD(%)	:UNK-011 Corr.Conc.(ppm) 2.59 2.59 0.00 0.00	Sample Name:C3 Conc.(ppm) 2.59 2.59 0.00 0.00	ABS REF 0.6063 C 0.8287 0.6063 0.8287 0.0000 0.0000 0.00 0.00
Sample ID No. 1 Mean SD RSD(%)	:UNK-012 Corr.Conc.(ppm) 2.29 2.29 0.00 0.00	Sample Name:04 Conc.(ppm) 2.29 2.29 0.00 0.00	ABS REF 0.5357 C 0.5357 O.9302 0.0000 0.0000 0.000 0.0000 0.00 0.000

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Detail Table

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	Analysis M Analysis M Comment	Mode :Flame/Autosan Name :Studies of Zu :Diana Singga	mpler n(II) ion adsorp r	tion by rice husk	
	Meas. Date	e :10/11/04 3:4	6 PM		
	Sample ID No. 1 Mean SD RSD(%)	:STD1 Corr.Conc. 	Sample Name: Conc.(ppm) 0.00 	ABS REF -0.0001 -0.0059 -0.0001 -0.0059 0.0000 0.0000 0.00 0.00	
	Sample ID No. 1 Mean SD RSD(%)	:STD2 Corr.Conc. 	Sample Name: Conc.(ppm) 0.20 	ABS REF 0.0455 0.0118 0.0455 0.0118 0.0000 0.0000 0.00 0.000	
	Sample ID No. 1 Mean SD RSD(%)	:STD3 Corr.Conc. 	Sample Name: Conc.(ppm) 0.40 	ABS REF 0.0982 0.0205 0.0982 0.0205 0.0080 0.0000 0.000 0.0000 0.00 0.000	
	Sample LD No. 1 Mean SD RSD(%) Coefficie Corr.Coef	:STD4 Corr.Conc. nt :K3= K2= K1=2.926571E-0 K0=-8.580001E- :0.9961	Sample Name: Conc. (ppm) 0.80 01 003	ABS REF 0.2318 0.0391 0.2318 0.0391 0.0000 0.0000 0.000 0.000	
				• • •	
	Sample ID No. 1 Mean SD RSD(%) Sample ID No. 1	<pre>:UNK-001 Corr.Conc.(ppm) 1.84 1.84 0.00 0.00 </pre>	Sample Name:B4 Conc. (ppm) 1.84 1.84 0.00 0.00 Sample Name:A5 Conc. (ppm) 2.17	ABS REF 0.5313 C 0.0868 0.5313 0.0868 0.0000 0.0000 0.00 0.00 ABS REF 0.6254 C 0.1066	
	Mean SD RSD(\$}	2.17 0.00 0.00	2.17 0.00 0.00	$0.6254 ext{ 0.1066} \\ 0.0000 ext{ 0.0000} \\ 0.00 ext{ 0.00} \\ 0.00 ext{ 0.00} \\ 0.00 ext{ 0.00} $	
•	Sample If No. 1 Mean SD RSD(%)	D :UNK-003 Corr.Conc.(ppm) 1.93 1.93 0.00 0.00	Sample Name:B5 Conc.(ppm) 1.93 1.93 0.00 0.00	ABS REF 0.5570 C 0.0920 0.5570 0.0920 0.0000 0.0000 0.00 0.000	
	Sample II No. 1	Corr.Conc.(ppm) 2,42	Sample Nam 44 05 Conc.(ppm) 2.42	ABS REF 0.7001 C 0.1287	

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Mean	2.42	2.42	0.7001	0.1287
SD .	0.00	0.00	0.0000	: 0.0000
RSD(%)	0.00	0.00	0.00	0.00
Sample ID	:UNK-005	Sample Name:A6		
No.	Corr.Conc. (ppm)	Conc. (ppm)	ABS	REF
1	2.86	2.86	0.8298	C'0.1851
Mean	2.86	2.86	0.8298	0.1851
SD .	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00
Sample ID	:UNK-006	Sample Name: B6		
NO.	Corr.Conc. (ppm)	Conc. (ppm)	ABS	REF
39 ¹ 1	2 41	2 41	0_6962	C 0.1271
Mean	2 41	2 41	0 6962	0.1271
SD	0.00	0.00	0.00002	0.0000
	0.00	0.00	0.0000	0.0000
Kon (s)	0.00	0.00	0.09	0.00
Sample ID	:UNK-007	Sample Name:C6		
No.	Corr.Conc.(ppm)	Conc. (ppm)	ABS	REF
1	2.10	2.10	0.6066	C ⁰ 0.1027
Mean	2 10	2 10	0.6066	0.1027
SD	0.00	0.00	0.0000	0,000,0
	0.00	0.00	0.0000	0.000
VOD (s)	0.00	0.00	0.00	0.00

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