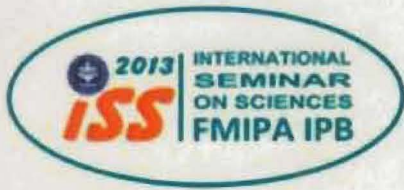


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PROCEEDINGS

INTERNATIONAL SEMINAR ON SCIENCES 2013

"Perspectives on Innovative Sciences"

FACULTY OF MATHEMATICS AND NATURAL SCIENCES,
BOGOR AGRICULTURAL UNIVERSITY
IPB International Convention Center
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Bogor Agricultural University

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FOREWORD

The International Seminar on Sciences 2013, which had the main theme "Perspectives on Innovative Sciences", was organized on November 15th -17th, 2013 by the Faculty of Mathematics and Natural Sciences, Bogor Agricultural University. This event aimed at sharing knowledge and expertise, as well as building network and collaborations among scientists from various institutions at national and international level.

Scientific presentations in this seminar consisted of a keynote speech, some invited speeches, and about 120 contributions of oral and poster presentations. Among the contributions, 66 full papers have been submitted and reviewed to be published in this proceeding. These papers were clustered in four groups according to our themes:

- A. Sustainability and Science Based Agriculture
- B. Science of Complexity
- C. Mathematics, Statistics and Computer Science
- D. Biosciences and Bioresources

In this occasion, we would like to express our thanks and gratitude to our distinguished keynote and invited speakers: Minister of Science and Technology, Prof. Manabu D. Yamanaka (Kobe University, Japan), Prof. Kanaya (Nara Institute of Science and Technology, NAIST, Japan), Prof. Ken Tanaka (Toyama University, Japan), Emmanuel Paradis, PhD. (Institut de Recherche pour le Développement, IRD, France), Prof. Dr. Ir. Rizaldi Boer, MS (Bogor Agricultural University), and Prof. Dr. Ir. Antonius Suwanto, M.Sc. (Bogor Agricultural University).

We would like also to extend our thanks and appreciation to all participants and referees for the wonderful cooperation, the great coordination, and the fascinating efforts. Appreciation and special thanks are addressed to our colleagues and staffs who help in editing process. Finally, we acknowledge and express our thanks to all friends, colleagues, and staffs of the Faculty of Mathematics and Natural Sciences IPB for their help and support.

Bogor, March 2014

The Organizing Committee

International Seminar on Sciences 2013

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The Use of Activated Carbon from Bintaro Fruit-Shell (*Cerbera manghas*) as an Adsorbent to Increase Water Quality

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Abstract

Bintaro seed is utilized widely as an alternative raw material to make biodiesel and to produce Bintaro fruit shells. In this research, the Bintaro fruit shell was used as an alternative raw material to make an activated carbon. Carbon activation were relied on 2 (two) factors, chemical activation (H_3PO_4 concentrate) and physics activation (time of water vapor steam). Activated carbon as quality indicator was characterized using Indonesia National Standard (SNI 06-3730-1995). The best activated carbon is produced from the shell treated with 15% H_3PO_4 , water vapour steamed in 90 minutes with 9.98% moisture content, 9.16% volatile matter, 12.45% ash content, 78.4% fixed carbon, absorption on 784.498 mg/g iodine, absorption of 17.73% benzene, and absorption of 127.705 mg/g methylene blue. The mechanism used for adsorption was Langmuir isotherm with 0.9691 linearity. The activated carbon was capable to reduce up to 100% and 86.94% iron and manganase concentrate respectively.

Keywords: activated carbon, *Cerbera manghas*, isotherm absorption

I. INTRODUCTION

Water is very important for the survival of living beings. The degradation of water quality makes it being nonfunctional. This problem can be resolved using an activated carbon.

The quality of activated carbon is based on its ability to adsorb the iodine. The minimum value of iodine to be adsorb by the adsorbent is regulated by the Indonesian National Standard (SNI)[1] is 750 mg/g. While the American Society for Testing Materials (ASTM D4607-94) and Japan Industrial Standards (JIS) require the value to be 1000 mg/g.

Activated carbon can be made from carbonized organic materials such as wood and brown coal [2]. Raw materials used for commercial activated carbon in Indonesia are generally from coconut shell, while in America and Japan they are generally made from coal. Commercial activated carbon raw materials that have met the ASTM and JIS are teaks. The use of teak is limited depend on the purpose of their utilization or categorized as an unrenewable resources. In this research the activated carbon was made from bintaro fruit-shells (*Cerbera manghas*) for their availability as abundant biomass waste because bintaro trees produce fruit all year round and do not require specific treatment [3] . Shell yield from Bintaro fruit is 67.36 % of all biodiesel production process [4] . Bintaro fruit-shells contain fiber and lignocellulosic which have similarity in nature with coconut shell [5] . Thus, this research aims to utilize Bintaro fruit-shells as an alternative raw material in the manufacture of activated carbon that can be used as an adsorbent of metals at water wells to improve water quality.

II. MATERIALS AND METHOD

Materials

The materials used were Bintaro fruit-shells from Bogor, commercial activated carbon which is made from coconut shell, H_3PO_4 solution, iodine, natrium thiosulphate, starch, blue methylene, benzene, metal standard solution of manganese (Mn), and wells' waters (Dramaga, Bogor). Instrumentation used in this research were modified drum kiln, retort activated carbon, oven, desiccator, fine sieve (100 mesh), porcelain cup, analytical balances, shaker, visible-ultraviolet spectrophotometer (UV-VIS) Shimadzu UV type - 1700, scanning electron microscope (SEM) EVO type ZEUS-50, Atomic Absorption spectroscopy (AAS) 7000 Shimadzu type, pH-meters, and glass equipments.

Method

Carbonization

Bintaro fruit-shells were inserted into the kiln drum. Then, it was ignited by burning the air hole with the aid of bait sticks. When the raw materials started to burn, the kiln air hole was closed and chimney was installed. The carbonization process was considered complete when the smoke out of the chimney was thinning and bluish, then the kiln was cooled for 24 hours.

Activated Carbon

Bintaro fruit-shells' carbon in granular form were soaked in different concentration of H_3PO_4 solution (5%, 10%, and 15%) for 24 hours and then washed, drained, and air dried. Moreover, activated carbon with and without activation of H_3PO_4 was put into a retort with a capacity of 0.3 kg and was heated at a temperature of 750 °C. After the retort

reached the required temperature, the water vapor was flow for 0 minutes (without steam), 60 minutes, or 90 minutes, to give 12 types of activated carbon that can be seen in Table 1.

Table 1 Modification of activated carbon treatment

| Chemical activation (concentration of H ₃ PO ₄ , %) | Physics activation (time of water vapor steam, minutes) | | |
|---|--|----|----|
| | 0 | 60 | 90 |
| | 0 | A1 | A2 |
| 5 | B1 | B2 | B3 |
| 10 | C1 | C2 | C3 |
| 15 | D1 | D2 | D3 |

Yield (SNI 1995)

The formed activated carbon (sample) was weighed and compared with the carbon weight. Yield was calculated by the formula:

$$\text{Yield} = \frac{\text{Sample weight}}{\text{carbon weight}} \times 100\% \quad (1)$$

Characteristics of Activated Carbon Water Content

A total amount of ± 1.00 g sample was weighed in a porcelain dish with a known dry mass, then it was oven-dried at 105 °C for 3 hours. After cooling in a desiccator the sample was weighed. The drying and weighing process were repeated every hour until a constant weight is obtained. The analysis was conducted in two replications. Water content was calculated by the equation:

$$\text{Water content} = \frac{(a-b)}{a} \times 100\% \quad (2)$$

a = initial mass weight (g)

b = final mass weight (g)

Volatile Matter Content

A total amount of ± 1.00 g sample was weighed in a porcelain dish with a known dry mass. Then it was heated in an electric furnace at a temperature of 950 °C for 10 min, cooled in a desiccator, and weighed. The dish is closed as tight as possible. The analysis are conducted in two replications. Volatile matter content was calculated by the equation:

$$\text{Volatile Matter Conten} = \frac{(a-b)}{a} \times 100\% \quad (3)$$

Ash Content

A total amount of ± 1.00 g sample was weighed in a porcelain dish with a known dry mass. Cup containing the sample was placed in an electric furnace at a temperature of 700 °C for 6 hours. After that, it was cooled in a desiccator and weighed. Drying and weighing were repeated every hour until a constant weight was obtained. The analysis was conducted in two replications. Ash content was calculated by the equation:

$$\text{Ash Content} = \frac{\text{Sample weight}}{a} \times 100\% \quad (4)$$

Bonded carbon content

Carbon in activated carbon is a result of the composing process / pyrolysis besides the ash (inorganic substances) and volatile matter (essential substances are still present in the carbon pores). The definition is only an approachment (ISO 1995).

$$\text{Bonded carbon content} = 100\% - (u + z) \quad (5)$$

u = ash content (%)

z = levels of volatile matter (%)

Iodine Adsorption

A total amount of ± 0.25 g samples were oven dried for 1 hour then placed in a 250 mL Erlenmeyer flask. To this flask, 25 mL of 0.1 N iodine solutions was added, then the Erlenmeyer immediately closed and shaken for 15 minutes. Any suspension that formed in the solutions was filtered away. Then, 10 mL of filtrate was pipetted into the Erlenmeyer and directly titrated with a solution of 0.1 N Na-thiosulfate until yellow color was appeared. After adding a few drops of 1% starch, titration continued until the blue color was completely disappeared. This analysis was performed in two replications. Determination of the iodine adsorption was done by using the following equation:

$$Q_i = \frac{\left\{10 - \frac{B \times C}{D}\right\} \times 12.693 \times 2.5}{a} \quad (6)$$

Q_i = iodine adsorption (mg/g)

B = the volume of Na-tiosulfat solution (mL)

C = Na-tiosulfat normality (N)

D = iodine normality (N)

12.693 = a total amount of iodine equivalent 1 mL Na₂S₂O₃ 0.1 N solution

Benzene Adsorption

A total amount of ± 1.00 g samples are weighed into a petri dish which has been known of their dry mass. The plate is then put into a desiccator that has saturated by a benzene vapor for 24 hours to allow accomplishment of adsorption equilibrium. Moreover, activated carbon is weighed

again. But the petri dish should left in the air for 5 minutes before it is weighed to remove benzene vapor attached to the cup. This analysis are performed in two replications. Determination of benzene adsorption is using the following equation:

$$\text{Benzene adsorption} = \frac{(h-m)}{m} \times 100\% \quad (7)$$

m = mass of activated carbon before adsorption
 h = mass of activated carbon after adsorption

Methylene Blue Adsorption

A total amount of 0.25 g sample are put into a 25 mL Erlenmeyer and also added by 1200 ppm blue methylene, then shaken for 30 minutes and filtered. The filtrate is taken for 1 mL and then inserted into 100 ml pumpkin drinks. After that, the absorbance is measured by UV-Vis spectroscopy at a wavelength of 664 nm. This analysis are performed in two replications. Determination of blue methylene adsorption is using the following equation:

$$\text{Blue methylene adsorption} = \frac{V \times (C_0 - C_a) \times f_p}{a} \quad (8)$$

V = the volume of methylene blue (L)
 C_0 = initial concentration of methylene blue (ppm)
 C_a = final concentration of methylene blue (ppm)
 f_p = dilution factor

Morphology analysis of Activated Carbon Using SEM

The analysis using Scanning Electron Microscope (SEM) was aimed to see the surface morphology of activated carbon before and after the application. Activated carbon was inserted into the SEM tool to be observed its pore diameter on the surface.

Adsorption Test and Adsorption Isotherms

Adsorption test was used to measure the effectiveness of the adsorption power of activated carbon from bintaro fruit-shells against a standard solution of manganese with various concentrations ranging from 30, 40, 50, and 60 mg / L. All treatments were done in 2 replications. A total amount of 0.50 g best activated carbon was added to 50 mL standard solution and then shaken using a shaker for 60 minutes. Each solution was filtered by a coarse filter paper. Standard solution concentration were measured before and after treatment using Atomic Absorption Spectroscopy (AAS). Mechanism of adsorption was calculated by Freundlich and Langmuir isotherm models.

Application Test

The quality of best activated carbon was tested by cleaning and purifying well's water against the heavy metals content contained. The water quality was tested before and after the purification by analyzing its color, pH, and the content of heavy metals.

III. RESULTS AND DISCUSSION

Yield of Activated Carbon

Carbon activation process was done by chemical modification (H_3PO_4) and physical process (steam water vapor). The activation process reduced the amount of carbon as tar, organic acids, and hydrocarbons which were initially present on the surface of the carbon so that the pores on the surface of activated carbon get bigger [6]. This was proved for the activation process using steam vapor, the longer the process was treated with steam vapor, the smaller the yield resulted (Fig. 1), which was caused by the reaction between carbon with water vapor (oxidizing gas) known as gasification reaction [2]. This reaction produced CO_2 and H_2 as shown by the following reaction

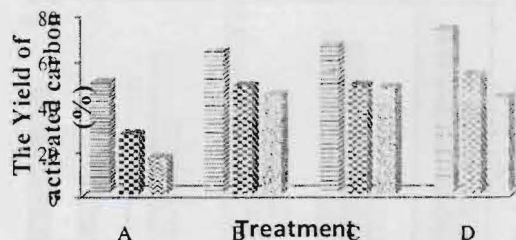
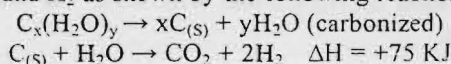


Fig.1. The yield of activated carbon with various treatment based on the concentration of H_3PO_4

- = without H_3PO_4 , A
- = H_3PO_4 5%, B
- = H_3PO_4 10%, C
- = H_3PO_4 15%, D
- ▨ = without steam water vapor, 1
- ▨ = steam water vapor for 60', 2
- ▨ = steam water vapor for 90', 3

On the other sides, using H_3PO_4 as activator tends to increase the yield of activated carbon because H_3PO_4 reduces the reaction speed in the oxidation process which means that H_3PO_4 functioned as a carbon protector at a given temperature [7]. Using of H_3PO_4 as activator can also reduce the yield of activated carbon due to the degradation of the micropore amount as the impact of broken wall between the pores to give the mesopore or macropore [8]. This result can be seen for the activated carbon with D3 treatment [Fig.2].

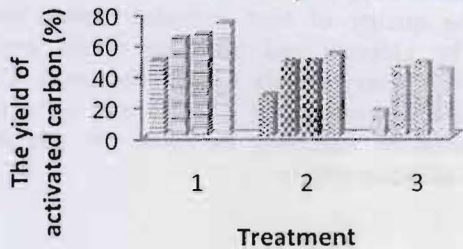


Fig.2. The yield of activated carbon of various treatment based on the time of steam water vapor

Characteristics of Activated Carbon

Water Content

Water content resulted from Bintaro fruit-shells were between 7.38-14.69%, the value that are still accepted by the Indonesia National Standard which was 15% maximal of water content for activated carbon. The activation using steam water vapor tends to increase water content (Fig.3) resulted from the hot water vapor trapped in the structure of hexagonal shaped activated carbon [9]. However, this condition was offset by the effects of dehydration of H_3PO_4 that will lower the water content.

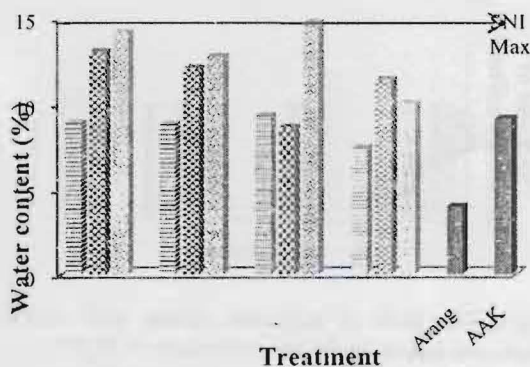


Fig. 3. Water content of activated carbon of various treatment based on the concentration of H_3PO_4

Volatile Matter and Ash Content

Volatile matter content of the activated carbon from bintaro fruit-shells ranged between 8.73-24.89% which was meet the SNI maximum value of 25%. Meanwhile, the ash content ranged from 8.31-19.86%, while the maximum value is 10% required by ISO. So that only a small percentage of ash content values are SNI eligible.

The activation process, both physical and chemical, tend to reduce the levels of volatile matter due to an increase in volume and surface size of the pore due to initial carbonization at temperature of 400-500 °C [10], and the exchange of OH groups on the raw material with PO_4 of H_3PO_4 can assist in breaking down the non-carbon compounds [11]. The higher the concentration of H_3PO_4 , the lower the volatile matter content

(Fig.4). However, the opposite can happened if H_3PO_4 added to the carbon permeated and coated, causing it to be protected from heat, also from the sulfur and nitrogen compounds. The content of volatile matter on carbon was larger than the activated carbon, because there was no non-carbon compound decomposed in the carbon.

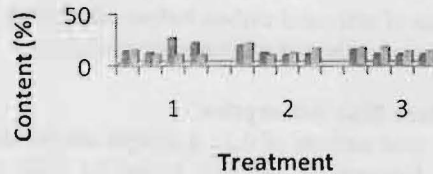


Fig.4. Volatile matter and ash content of activated carbon based on the time of steam water vapor.

Bonded carbon content

Bonded carbon content of the activated carbon from bintaro fruit-shells was ranged between 62.44-81.26% with the SNI minimum requirement value of 65%. The higher the levels of carbon bonded, the higher carbon purity on the activated carbon, and the cleaner the activated carbon resulted, which in turn increasing the adsorption capacity of the activated carbon (Fig.5).

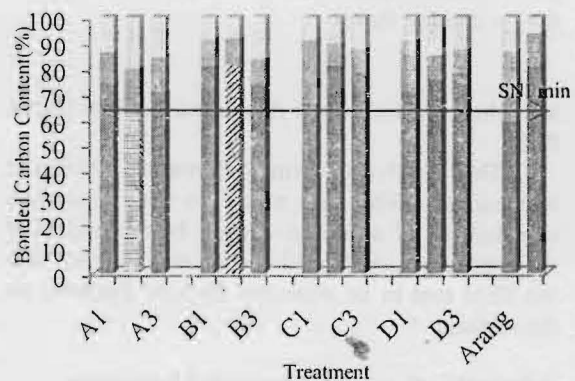


Fig.5. Bonded carbon content, volatile matter content, and ash content (Max, min)

The adsorption of Benzene, Methylene Blue, and Iodine

The adsorption of benzene, methylene blue, and iodine by the activated carbon were in the range of 8.1-17.73%, 21.9-127.71 mg / g, and 322.87-784.50 mg / g, respectively. Those values are smaller than the adsorption of benzene by activated carbon activated with KOH, which is 17.97-24.19% [12]. The longer the exposure with steam water vapor, the polarity of activated carbon is increasing as well (Fig.6) because this process will form an active groups such as carboxyl, quinone, hydroxyl, carbonil, carboxylic

anhydride, or lactone [10]. The polar properties of activated carbon facilitate the interaction between activated carbon and water.

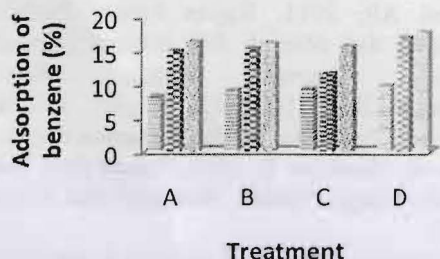


Fig.6. Adsorption of benzene by the activated carbon

Increasing the H_3PO_4 concentration will also increase the adsorption of activated carbon (Fig.7). Carbon which was activated with H_3PO_4 tends to produce the polar activated carbon, because H_3PO_4 decomposed into material such as P_2O_5 that can be attached and bounded at the edges of the pores of activated carbon to yield a more polar adsorbent [11]. Activated carbon resulted from Bintaro fruit-shell has better quality than the AAK

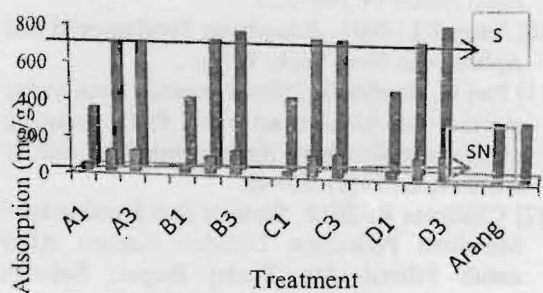


Fig.7. Adsorption of methylene blue and iodine

The Adsorption of Metals by Activated Carbon

Based on the analysis of heavy metals content in well water, it was obtained that well water only contains heavy metals of Mn and Fe (Fig.8).

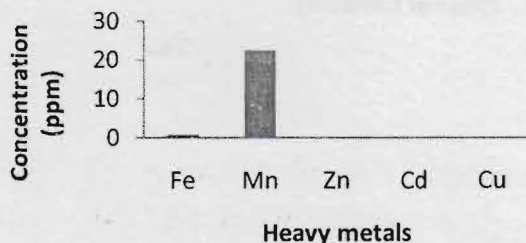


Fig.8. The heavy metals content in well water

The ability of activated carbon from Bintaro fruit-shells to adsorb metal is high enough. This was proved by ferrous metals level contained in the

well water, which was perfectly adsorbed and meet the requirement for drinking water [13]. In addition, the capacity of activated carbon in absorbing the manganese is large enough, with the value of 86.94%. However, the levels of manganese in well water do not meet the drinking water requirements after it was treated with activated carbon (Table 2).

Metal adsorption also resulted in changes of pH of the water from the initial conditions of acidic to 7.58. The pH still meets the requirements for drinking water, which is still in neutral pH range. Increase in pH value was due to the interaction between the active site on activated carbon with metal ions from well water, M^{2+} , resulted in the formation of complex compounds. In addition, there were physical changes of the well water, where the color was ranging from cloudy yellow to colorless.

Table 1 Well water quality

| Parameter | Observation | | Metal Adsorption Capacity (%) | Drink water standard [13] |
|-----------|---------------|-----------|-------------------------------|---------------------------|
| | Before | After | | |
| Fe (mg/L) | 0.6684 | 0.0000 | 100 | 0.3 |
| Mn (mg/L) | 22.3492 | 2.9198 | 86.94 | 0.1 |
| pH | 6.00 | 7.58 | none | 6.5–8.5 |
| Color | Cloudy yellow | Colorless | none | Colorless |

Description: Detection limit of AAS is 0.0001–0.0002 ppm

The adsorption of metal by activated carbon from bintaro fruit-shells was also indicated by the closing of high pore diameter in the range of 6031–17021 μm (Fig.8a). Adsorption mechanism of heavy metals in the water was following the Langmuir isotherm (Figure 9). It means that there is a formation of a single layer of adsorbate molecules on the adsorbent surface and the active site is homogen so that the absorption energy is the same on the whole active sites. Also, there is no adsorbate intermolecular interaction, and the bonds occurred between the adsorbate-adsorbent is chemisorpsi [14]

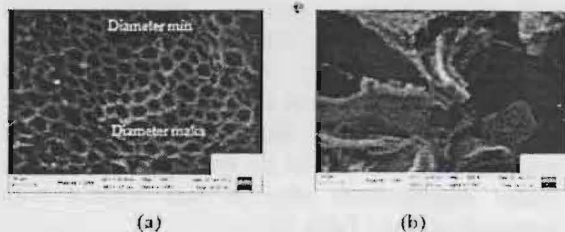


Fig.8. Topography of activated carbon- before (a) and after (b) applied in well water

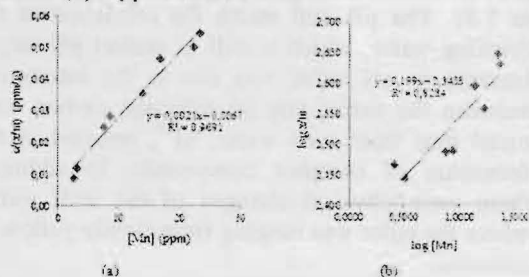


Fig.9. Isotherm adsorption (a) Langmuir and (b) Freundlich of Mn metals

IV. CONCLUSION

The activated carbon from Bintaro fruit shell which was activated with 15% of H_3PO_4 and a 90-minute steam vapor exposure can be used as an adsorbent in water quality improvement with metal adsorption capacity of Mn and Fe respectively, 86.94% and 100%, and qualified as SNI technical activated carbon. The activated carbon quality was 2.5 times higher than the quality of commercial activated carbon made from coconut shell based on the iodine adsorption of 784.498 mg/g. However, its quality has not passed the ASTM and JIS standards therefore do not meet the export quality.

ACKNOWLEDGEMENT

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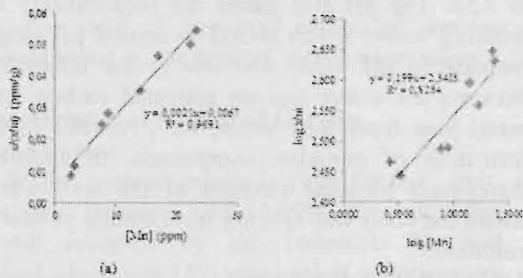


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