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Removal of Multi-heavy Metals from Simulated Wastewater using Hybrid Modified Carbonized Palm Shell and Rice Husk Adsorbents

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Abstract— **It necessary to remove heavy metals from wastewater in order to comply with environmental regulations and for sake of human health and safety. The purposes of the present study was to prepare hybrid modified adsorbent from the carbonized palm shell and rice husk by physical treatment for the removal of 13 multi heavy metal component from simulated wastewater by single bed adsorption column. The Metals being studied are Cadmium, Chromium, Copper, Iron, Magnesium, Manganese, Lead, Lithium, Molybdenum, Nickel, Antimony, Vanadium and zinc. Six types of adsorbents were used for metal removal which are (100% BRH) adsorbent; (100% CPS) adsorbent , and four hybrids adsorbent prepared by mixing the blended rice husk and carbonized palm kernel shell with mixing ratios 20% , 40% , 60% , 80% wt Blended Rice Husk . The obtained results of the individual area under the graph analysis showed that cadmium, copper, lithium; vanadium elements were strongly adsorbed by hybrid adsorbents with achieved removal efficiency of 99.95%, 99.97%, 31.23%, and 89.58% with least area under the graph values of 3294.65, 2015.52, 118.231, and 115.18 respectively. As a conclusion, the mixing of blended rice husk and carbonized palm shell as hybrid adsorbent contributed to enhance the adsorption performance of cadmium, copper, lithium, vanadium, elements which demonstrated by less area under the graph values compared with pure carbonized palm shell and pure rice husk adsorbents.**

Index Terms: **Adsorption, multi-heavy metals, wastewater, cabonized palm shell, rice husk .**

I. INTRODUCTION

Concerns about environmental protection have increased due to the technical development which keeps in changing, producing industrial product, as well as waste. Manufacturing industries have played an important role for economic growth in major countries. This sector provides services and product for better way and quality of living. However, rapid changes in industrialization produce vast volume of waste which cause harm and deteriorate the environment and ecosystem. Pollutants from heavy metals are widely discharged in the wastewater from industries such as cadmium (Cd), chromium (Cr), lead (Pb), copper (Cu), manganese (Mn), zinc (Zn) as well as mercury (Hg). These elements are very toxic and harmful to living organisms by lowering the reproductive success; prevent proper growth and development, and even causing death (Alturkmani, 2004). It is therefore necessary to remove heavy metals from wastewater in order to comply with environmental regulations and for the sake of human health and safety. There are many technologies have been developed for treatment and purification of waste water including chemical precipitation, solvent extraction, oxidation, reduction, dialysis/electro dialysis, electrolytic extraction, reverse osmosis, ion-exchange, evaporation, cementation, dilution, adsorption, etc. (Mohan and Singh, 2002).Among such treatment methods , Adsorption process is found to be the most suitable technique to remove pollutants from wastewater. It is mostly preferred due to the high reliability, energy efficiency, design flexibility, technological maturity and ability to regenerate the exhausted adsorbents (Mohan and Pittman, 2006). Activated carbon has been identified as the most widely used adsorbent for adsorption process to remove various types of metals. This is due to its higher surface area and porous structure, which then lead to higher adsorptivity. Despite of these advantages, the manufacturing cost of adsorption process is expensive due to high cost of activated carbon and regeneration process. It has been identified that, 10 – 15% loss occurred during regeneration of activated carbon adsorbent (Hashem, 2007). Consequently, such drawbacks limit its large scale application. Furthermore, Abdel Wahab et al. (2005) identified that, laboratory preparation of activated carbon has been impractical due

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to number of limitations such as pore blocking and combustion at higher temperature. Thus based on this situation, it is necessary to seek for other natural and inexpensive materials which have strong capability to adsorb metals in wastewater streams. Compared to conventional adsorbents, the natural adsorbents pose a lot of advantages; they are inexpensive, effective, readily and locally available, technical feasible and widely applicable in engineering. Accordingly, one of the waste materials in the world as reported in the literature with the most potential is rice husk, due its high availability (chuah et al., 2005) . In an attempt to address the cost problems associated with commercial adsorbents; the use of abundant, locally available, low cost adsorbents derived from agricultural wastes which are oil palm shell and rice husk are proposed in this study. However, the application of untreated agricultural wastes material as adsorbents can also bring several problems such as low adsorption capacity, (Gaballah et al., 1997; Nakajima and Sakaguchi, 1990). Therefore, theses materials need to be modified or treated before being applied for the decontamination of heavy metals. Most of the previous researchers focused on the removal of one type of elements by using agriculture waste materials such as, hexavalent chromium ion (Owlad *et al*., 2010), copper ions (Hossain *et al.*, 2011), chromium (Saueprasearsit, 2010), nickel (Totlani *et al.* 2012). There are very few studies on the removal of multi heavy metal from wastewater. In reality, there were many types of elements in wastewater and there is a lack of research on the removal of multi-metal from waste water. This research study was conducted to investigate the ability of produce a hybrid adsorbent from palm shell and rice husk and modified by means of physical treatment to use in treating multi-heavy metals from wastewater. The purpose of the present work was to prepare hybrid modified adsorbent from the carbonized palm shell and rice husk by physical treatment for the removal of multi elements from wastewater by single bed adsorption column.

II. MATERIAL AND METHODS

*A. Materials***:** Materials involved in this study include simulated wastewater as well as rice husk and Palm Kernel Shell adsorbents.

*B***.** *Methods*

B.1 Preparation of simulated wastewater

The simulated wastewater was prepared by mixing several chemicals, which were the iron (II) sulphate which obtained from Acros Company and cadmium chloride which was also purchased from Acros Company ; copper (II) nitrate trihydrate, lead nitrate, chromium (VI) oxide which were applied by Merck Company ; magnesium chloride which was obtained from Bendozen Company; manganese (IV) oxide which was purchased from Fischer Company , zinc sulphate and aluminum chloride which were obtained from Hmbg Company , 1g of each chemical was mixed in 250 mL of distilled water. The solution was stirred using a magnetic stirrer at 2000 rpm for 5 minutes to homogeneously mix. Then, all the prepared solutions were put together in plastic container, diluted with distillated water until the total volume reach 10L. Then the mixed solution was stirred at 2000 rpm for two hours using electric mixer and then final mixed solution was stored in a closed chemical container.

B.2. Preparation of Blended Rice Husk and Modified Carbonized Palm Kernel Hybrid adsorbents:

Raw Rice Husk was obtained from *Padi Beras Nasional Berhad* (BERNAS) Rice Milling in Sekinchan, Selangor, Malaysia. Palm kernel shells was obtained from the Palm Oil Mill Technology Center (POMTEC), Labu, Negeri Sembilan, Malaysia.Rice husk and palm kernel shell adsorbent were used to prepare the hybrid adsorbents. Firstly, The Raw Rice Husk was washed with distilled water and filtered by using GAST Diaphragm Vacuum Pump, prior to drying in MEMMERT Universal Oven at the temperature range of 105 – 110°C for 24 hours. Then Raw Rice Husk underwent a physical treatment where it was blended for size reduction by using Waring Commercial Laboratory Blender. Then, it was sieved to 150 – 250 µm size by using Retsch Mechanical Sieve Shaker. The blended and sieved rice husk was washed with distilled water by stirring at 600 rpm for 15 minutes. After that, it was filtered prior to drying as mentioned early. Grinded rice husk was termed as Blended Rice Husk. The sample was stored in closed plastic bottle. Secondly, the raw palm kernel shell was washed, filtered to remove dirt and impurities, and dried in Memmert Universal Oven at the temperature range of 105 – 110°C for 24 hours. The raw palm kernel shells underwent a physical treatment where they were carbonized at 600°C in a furnace for 5 hours then underwent size reduction by grinding using the Waring Commercial laboratory blender and sieved to the desired mesh size in a range of sizes of 150 to 250 µm . Grinded carbonized palm shell was termed as carbonized palm kernel shell. The sample was then stored in closed plastic bottle. Six adsorbents were prepared and used on single bed column adsorption experiments. Two of the adsorbents are pure blended rice husk (100% BRH); pure carbonized palm kernel shell (100% CPS) and other four hybrids adsorbent are prepared by mixing the blended rice husk and carbonized palm kernel shell with mixing ratios 20% , 40% , 60% , 80% wt Blended Rice Husk . The amounts of blended rice husk and carbonized palm kernel shell which used in preparing the above mentioned adsorbents are shown in Table 1.

Table 1. Amounts of Blended Rice Husk and Carbonized Palm Kernel Shell Used in Preparing the Hybrid Adsorbents

Adsorbent Composition	**Amount of Blended Rice Husk(g_m)**	**Amount of carbonized palm shell(g_m)**
100%RH	6	0
80%RH	4.8	1.2
60%RH	3.6	2.4
40%RH	2.4	3.6
20%RH	1.2	4.8
100% CPS	0	6

B.3 . Screening Analysis of Simulated Wastewater

Random concentration of multi-metal element in the simulated wastewater sample was analyzed and quantified by using Thermo Scientific Inductively Couple Plasma (ICP) Spectrometer. For this purpose, 8 mL of simulated wastewater sample was taken out from the 10 Liter plastic chemical container.

B.4. Characterizations of Carbonized Palm Shell and Rice Husk Adsorbents

The adsorption capacity is mostly influenced by the adsorbents, porosity, total surface area, internal pore volume and carbon content. In this study the adsorbents underwent only one type of characterization which is surface morphology analysis. Field Emission Scanning Electron Microscope (FESEM) was used to study the surface morphology of the rice husk and palm shell adsorbents. Specifically, FESEM studies were carried out by using a ZEISS SUPRA 40VP FESEM at an electron acceleration voltage of 1 kV and magnification of 1000 times.

B.5. Fixed Bed Adsorption Study

The objective of this set of experiment was to perform the adsorption performance in a single- layered fixed bed column to adsorb multi-metal element from simulated wastewater. The fixed bed adsorption column was constructed at the laboratory scale to study the adsorption performance of six different adsorbents to remove metals from simulated wastewater. A 250 ml burette (diameter 1.6 cm) was used as a column for packing. All the six physically treated adsorbents were applied by single bed column with the same amount. A cotton ball with a thickness of 1 cm is placed inside the burette glass column at the bottom up to 20 cm above the stopcock. The adsorbent with a weight of 6 gm was introduced into the column in small quantities. A test tube was placed at the end of column by the retort stand to collect the treated simulated wastewater. The burette was charged with simulated wastewater in the down flow mode manually. Simulated wastewater was discharged manually at a constant fixed flow rate at the atmospheric pressure. The treated wastewater samples were then collected at intervals until the volume for each sample is 8mL. The concentration of treated wastewater samples was measured by the ICP-OES. The experimental setup for single layer fixed bed column is presented in Figure 1. The experimental rigs were assumed as semi-batch because it involved continuous inlet and outlet wastewater flows and batch adsorbent displacement. The adsorption system inside the column is in atmospheric pressure. Inlet simulated wastewater flow rate, mass of adsorbent, and volume of treated wastewater at certain time interval were also fixed throughout the studies.

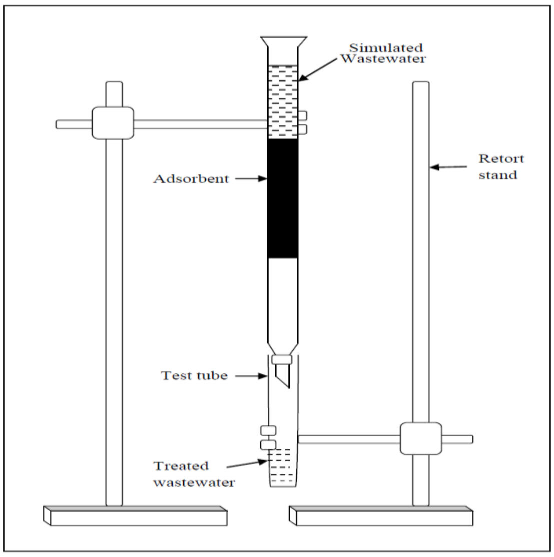

Figure 1*:* Experimental Setup for Single Bed Adsorption

B.6 .Data Analysis

B.6. 1 Area under the Graph Analysis

The treated wastewater samples from all experiments were analyzed for residual metals concentrations after adsorption by using Thermo Scientific ICP Spectrometer. Adsorption performance of all experiments was evaluated by area under the graph analysis using the trapezoidal rule. Since the number of interval during 180 minutes was different for every adsorbent, the non-uniform trapezoidal rule was adopted throughout the study. The equation used for non-uniform trapezoidal rule is presented in equation 1 below:

$$\int_a^b f(x)dx \approx \frac{1}{2}\sum_{k=1}^{N}\left(x_{k+1}-x_k\right)\left(f\left(x_{k+1}\right)+f\left(x_k\right)\right) \quad (1)$$

Where: a = value at first point of time, b = value at last point of time , N = number of interval, k = point of a value

Graphs of concentration of metal element, i (C_i) at contact time (t) for adsorption processes were plotted accordingly. Theoretically, integration is a process of measuring the area under the graph. It has been identified that, the adsorption performance of the column increases with decreasing area under the graph. The small area under the graph represents high adsorption capacity, while the large area under the graph represents low adsorption capacity.

B.6. 2 Removal Efficiency Analysis

Adsorption behaviors of multi – metal element towards each type of rice husk adsorbents were studied by evaluating percentage removal efficiency from Equation (2) below.

$$\text{Removal efficiency} = \left[\frac{C_0 - C}{C_0}\right] \times 100 \quad (2)$$

Where: C_0 = Initial concentration of metal element, C = Concentration of metal element after adsorption at any time

III. RESULTS AND DISCUSSION

A. Screening Analysis of Simulated Wastewater

The initial concentration of multi-metal element in simulated wastewater sample before adsorption was analyzed by ICP Spectrometer.The resulted concentrations of metals were Cadmium(Cd); 68.3002 ppm , Chromium(Cr); 45.109 ppm , Copper (Cu); 80.9177 ppm , Iron (Fe); 8.04997 ppm , Magnesium (Mg); 33.0103 ppm , Manganese (Mn); 0.786 ppm, Lead (Pb); 0.0304424 ppm; Lithium (Li) 0.98061 ppm; Molybdenum (Mo) 2.75201 ppm; Nickel (Ni) 0.0159597 ppm; Antimony (Sb) 0.114958 ppm; Vanadium (V)2.51915 ppm; Zinc (Zn) 29.6202 ppm .These results were then used as reference concentrations of multi-metal element for all adsorption studies. Furthermore, the screening analysis of simulated wastewater also indicated that, the wastewater sample was characterized by high toxicity of metals elements.

B. Surface Morphology Analysis of Rice Husk and Palm shell Adsorbents:

Micrographs of Rice Husk and Palm shell adsorbents are shown in Figures 2.0 until 5.0. Micrographs show considerable changes in morphology of rice husk and palm shell adsorbent after physical treatment.

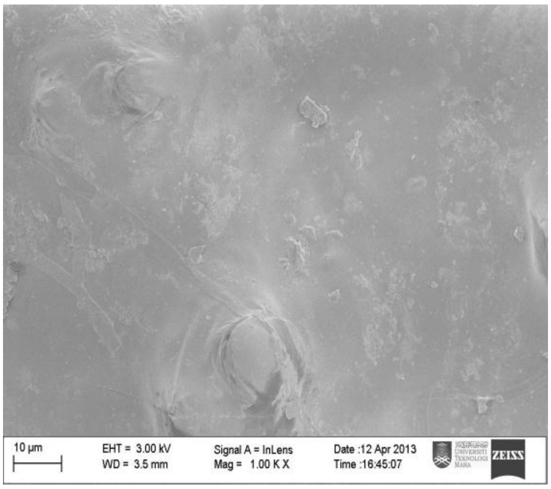

Figure 2: Micrograph of the Raw Rice Husk.

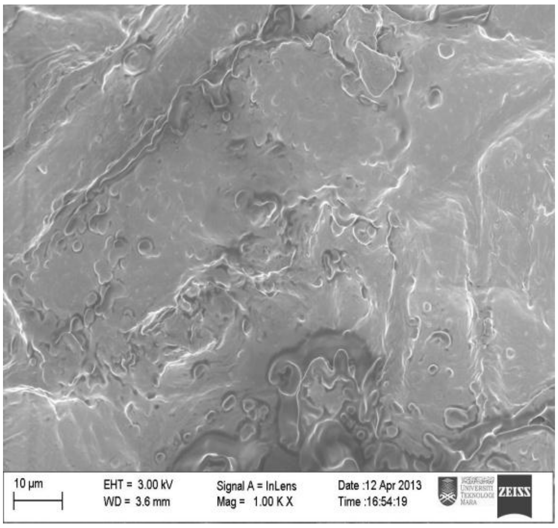

Figure 3: Micrograph of the Raw Palm Shell.

As shown in Figures 2, 3, it was observed that the pore on the surface is hardly seen, such that a thin layer exists on the surface and covers the pores. It seemed that the layer is comprised of volatile compound and moisture, which caused the pores to be hardly seen. The adsorption can still occur on the surface but in limited space.

Figure 4, 5 presents the micrograph of carbonized palm shell and blended rice husk .Upon carbonization, pores are seeable, pores are seeable clearly especially in palm shell micrograph, The thin layer appeared previously in Figure 2, 3 seems to disappear, due to the effect of carbonization and size reduction . The carbonization of palm shell produces pores and eliminates volatile compound, moisture and other elements to generate clearer surface and larger surface area for adsorption of multi-metal element. The surface of Blended Rice Husk was much rougher and highly heterogeneous than that of Raw Rice Husk, demonstrating the effect of size reduction. The heterogeneous surface of Blended Rice Husk provided more exposed surface area for adsorption of multi-metal element.

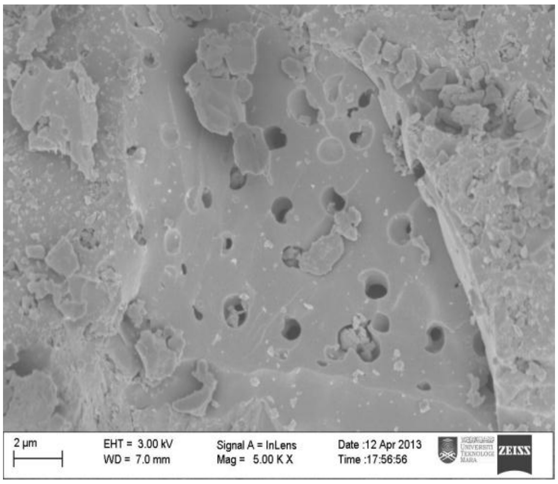

Figure 4: Micrograph of the Carbonized Palm Shell.

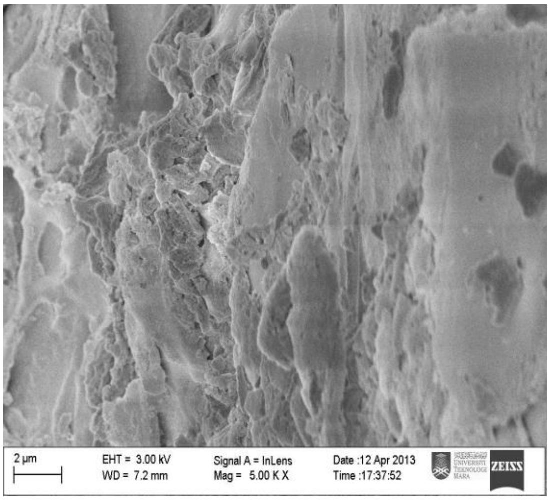

Figure 5. Micrograph of the Blended Rice Husk.

C. Results of Area under the Graphs and removal efficiency Analysis:

Area under the graphs analysis was performed to evaluate the adsorption performance of each experimental study. Thus, graphs of concentration of metal element *i* (C_i) in the treated wastewater at contact time (*t*) by different types of rice husk and palm shell hybrid adsorbents were plotted accordingly and the most important curves which showed high adsorption performance are depicted in Figures 6 until 15 for single layer bed adsorption. After plotting the graphs, the individual area under the curve for each metal element was determined by trapezoidal rule and the obtained results for all the elements are arranged in Table (2) swhich shown in appendix. In terms of removal efficiency after 180 minute of adsorption the obtained values of removal efficiency is presented in Table (3) in appendix .

As apparent in Table (2), the pattern of the area under the graphs for each metal element by different types of Rice Husk and Palm Shell Hybrid Adsorbents were varied without consistent pattern due to competition factors in multi-component adsorption. Based on the area under the curve values which results from adsorption studies in single layer bed, it can be observed that there were elements that can be removed with high adsorption capacity comparing with pure blended rice husk and pure carbonized palm shell by applying rice husk and palm shell hybrid adsorbents in single layer bed adsorption study which are cadmium, copper, lithium, vanadium, with achieved removal efficiencies of 99.95%, 99.97%, 31.23%, 89.58% respectively and this result was probably due to high interaction which generated between metal ion and hybrid adsorbent surface **.** Conversely, there were elements that can be removed with high adsorption capacity comparing with the rice husk and carbonized palm shell hybrid adsorbents by applying the pure carbonized palm shell and pure blended rice husk which are chromium, iron, nickel, antimony with achieved removal efficiencies of 95.44%, 99.98%, 37.43%, 100% respectively and this result was probably duo to metal ion attractive toward interaction with pure surface adsorbent as for iron toward pure rice husk adsorbent surface and chromium for pure carbonized palm shell adsorbent surface. On the other hand, there were elements can't be removed by all the adsorbents applied except pure carbonized palm shell with low capacity occasionally which are magnesium, manganese, molybdenum and this result might be due to low interaction between metal ion and adsorbent surface . According to the results of single layer bed adsorption studies, fast desorption of some metals like cadmium, copper, antimony and zinc occurred at the beginning of adsorption duo to low interaction between metal ion and adsorbent surface and as result , the concentration of the metal increased and adsorption capacity decreased. Future more, a desorption of other metals like nickel, lead, and antimony occurred at the end of adsorption time which might be duo to long time of adsorption, thus releasing the metal ion adsorbed into the wastewater , and as a result , the concentration of the metal increased which leaded to decrease the adsorption capacity. Moreover, the concentration of some metals like cadmium, antimony and zinc increased after awhile of adsorption and sometimes exceed the initial concentration of the metal ion; this result might be duo to the competition and inhibition by the other elements which is commonly exist in multi component adsorption. occurred.

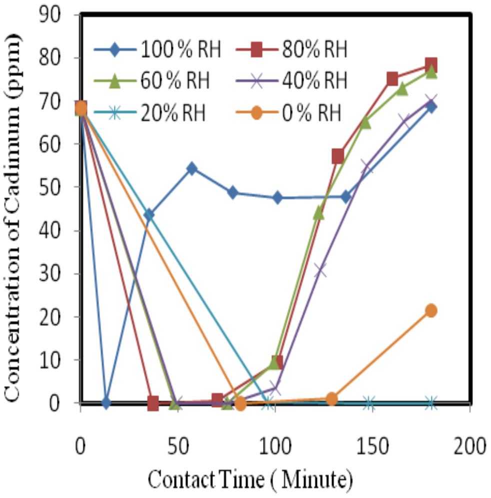

Figure 6. Adsorption of Cadmium by Physically Treated Hybrid Adsorbents.

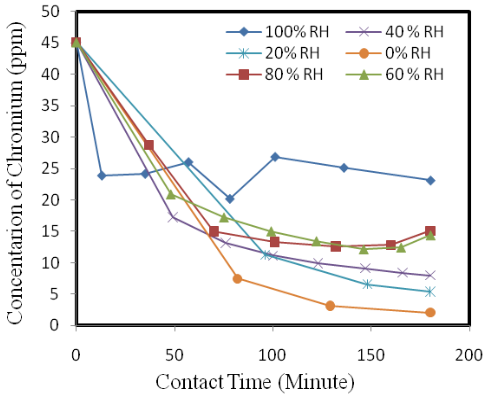

Figure 7. Adsorption of Chromium by Physically Treated Hybrid Adsorbents.

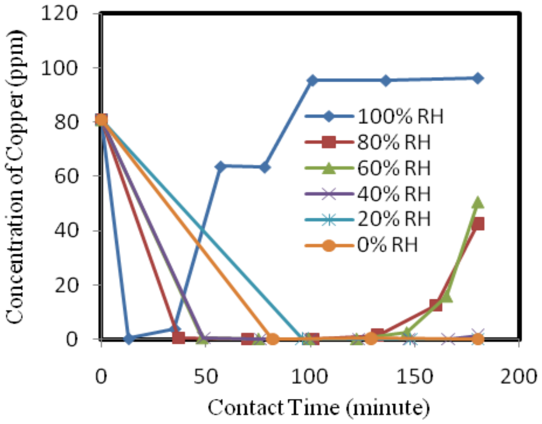

Figure 8. Adsorption of Copper by Physically Treated Hybrid Adsorbents.

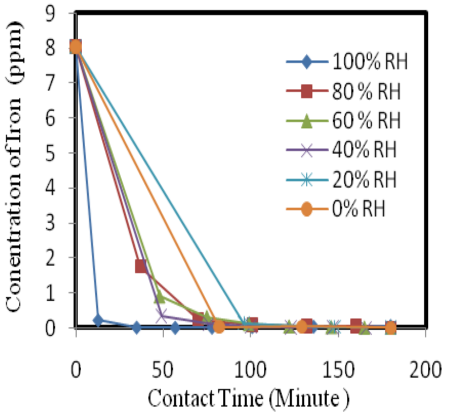

Figure 9. Adsorption of Iron by Physically Treated Hybrid Adsorbents.

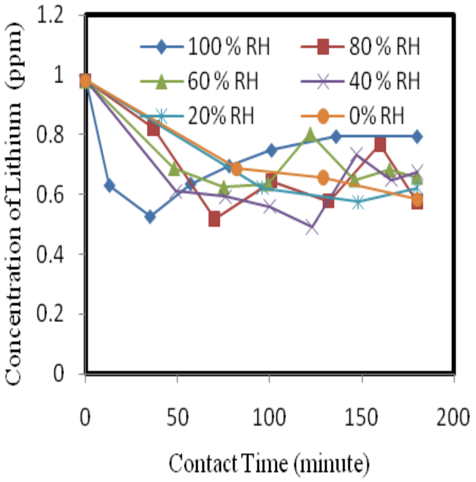

Figure 10 .Adsorption of Lithium by Physically Treated Hybrid Adsorbents.

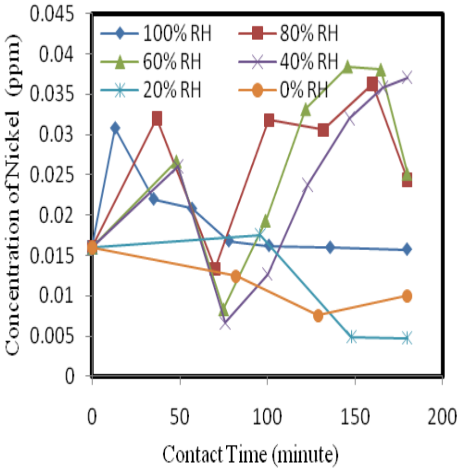

Figure 11. Adsorption of Nickel by Physically Treated Hybrid Adsorbents.

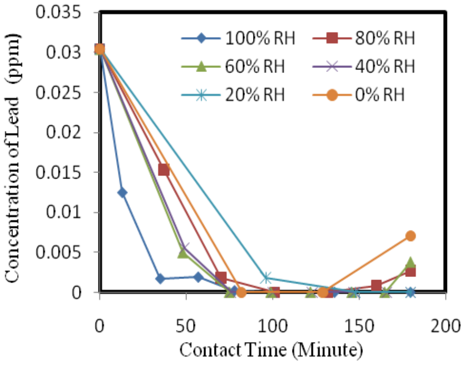

Figure 12. Adsorption of Lead by Physically Treated Hybrid Adsorbents.

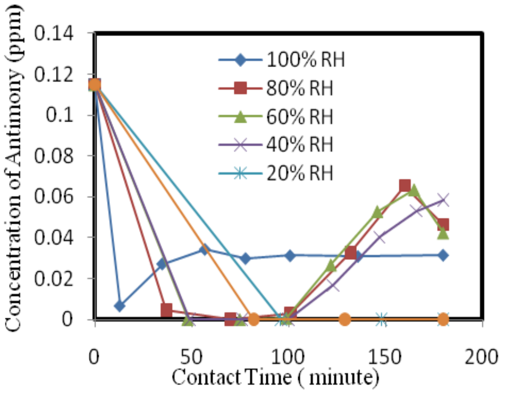

Figure 13. Adsorption of Antimony by Physically Treated Hybrid Adsorbents

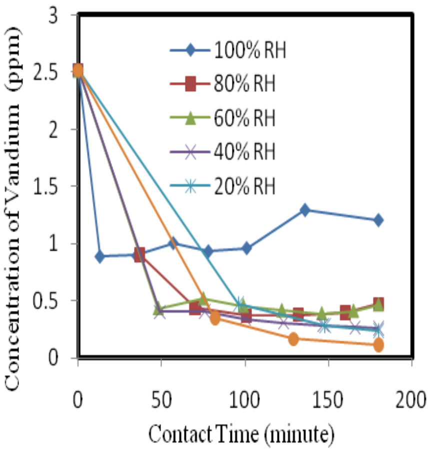

Figure 14: Adsorption of Vanadium by Physically Treated Hybrid Adsorbents.

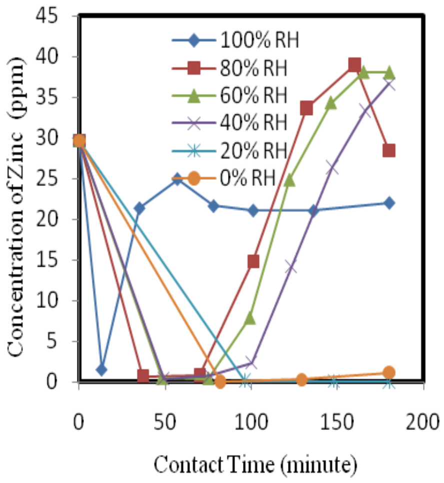

Figure 15: Adsorption of Zinc by Physically Treated Hybrid Adsorbents.

IV. CONCLUSION

As previously motioned in this study, Six types of adsorbent were produced , treated physically and used in removal of multi-metals from simulated wastewater , based on the obtained results of the adsorption performance of adsorption studies which indicated by individual area under the graph for each element , it can be conclude that mixing of blended rice husk and carbonized palm shell as hybrid adsorbent contributed to enhance the adsorption performance of some heavy metals like cadmium, copper, lithium and vanadium and which demonstrated by less area under the graph values compared with pure carbonized palm shell and pure rice husk adsorbents. On the other hand, It is important to point out that, the pattern of area under the graphs for each type of metal element by different mixing ratio of rice husk and palm shell hybrid and pure blended rice husk and carbonized palm shell adsorbents were varied without consistent pattern due to competition factors and roll up effects in multi-component adsorption. Thus for future study it is recommended to evaluate the adsorption performance of the studied adsorbents based on the total area under the graph for total removal of multi-metal element instead of individual area under the graph for each metal. Moreover, use of multi-layer fixed bed column can also be employed to evaluate the effect of number of beds on adsorption performance.

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APPENDIX

Table (2) Area Under the Graphs Values for Single Layer Bed Adsorption Studies by Different Types of Rice Husk and Palm Shell Hybrid Adsorbents.

Element	**Adsorbent**					
	Area under the curve					
	0% RH	**20%RH**	**40%RH**	**60%RH**	**80%RH**	**100%RH**
Cadmium	3400.84	*3294.65*	5233.36	6126.96	5852.79	8430.18
Chromium	*2539.42*	3362.97	2981.53	3561.24	3563.30	4528.42
Copper	3342.75	3901.41	2015.52	2662.59	2288.68	12046.96
Iron	334.365	398.382	215.093	240.088	224.078	60.74
Lithium	131.577	127.225	118.231	129.555	124.605	128.41
Magnesium	6518.93	7763.78	6683.93	6742.36	6746.84	6285.13
Manganese	122.84	324.61	717.70	896.00	1203.53	414.67
Molybdenum	212.64	593.32	1080.99	1333.08	1847.32	716.46
Nickel	2.077	2.34	3.94	4.49	4.84	3.39
Lead	1.42	1.59	0.956	0.9452	1.205	0.499
Antimony	4.71	5.51	5.36	5.94	5.37	5.67
Vanadium	136.88	171.56	115.18	129.06	128.97	199.68
Zinc	1257.17	1436.71	2523.05	3172.66	3268.79	3628.29
Total area	**18005.6**	**21384.12**	**21689.5**	**24999.0**	**25254.9**	**36442.87**

Table (3). Removal Efficiency Values of Metals from Wastewater in Single Layer Bed Adsorption Study

Elements	**Removal Efficiency(%) after 180 minute**					
	0%RH	**20%RH**	**40%RH**	**60%RH**	**80%RH**	**100%RH**
Cadmium	68.43	99.95	n/a	n/a	n/a	n/a
Chromium	95.44	87.97	82.25	68.03	66.59	48.76
Copper	99.92	99.97	98.15	37.42	47.32	n/a
Iron	99.98	99.80	100	99.88	99.89	99.25
Lithium	40.48	36.58	31.23	32.92	41.33	19.02
Magnesium	n/a	n/a	n/a	1.44	6.42	n/a
Manganese	n/a	n/a	n/a	n/a	n/a	19.66
Molybdenum	17.68	n/a	n/a	n/a	n/a	44.18
Nickel	37.43	70.16	n/a	n/a	n/a	1.37
Lead	76.79	100	100	87.56	91.16	100
Antimony	100	100	49.06	63.08	59.69	72.58
Vanadium	95.42	90.63	89.58	81.52	81.37	52.01
Zinc	96.21	99.83	n/a	n/a	4.07	25.66

Note: n/a refers to concentration of element exceeded its initial concentration.