

## Heavy metals removal from synthetic wastewater by a novel nano-size composite adsorbent

Y. B. Onundi; \*A. A. Mamun; M. F. Al Khatib; M. A. Al Saadi; A. M. Suleyman

Bioenvironmental Engineering Research Unit, Department of Biotechnology Engineering, Faculty of Engineering,  
International Islamic University Malaysia, Gombak, 53100 Kuala Lumpur, Malaysia

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**ABSTRACT:** The effects of varying operating conditions on metals removal from aqueous solution using a novel nano-size composite adsorbent are reported in this paper. Characterization of the composite adsorbent material showed successful production of carbon nanotubes on granular activated carbon using 1 % nickel as catalyst. In the laboratory adsorption experiment, initial mixed metals concentration of 2.0 mg/L  $\text{Cu}^{2+}$ , 1.5 mg/L  $\text{Pb}^{2+}$  and 0.8 mg/L  $\text{Ni}^{2+}$  were synthesized based on metals concentration from samples collected from a semiconductor industry effluent. The effects of operation conditions on metals removal using composite adsorbent were investigated. Experimental conditions resulting in optimal metals adsorption were observed at pH 5, 1 g/L dosage and 60 min contact time. It was noted that the percentage of metals removal at the equilibrium condition varied for each metal, with lead recording 99 %, copper 61 % and nickel 20 %, giving metal affinity trend of  $\text{Pb}^{2+} > \text{Cu}^{2+} > \text{Ni}^{2+}$  on the adsorbent. Langmuir's adsorption isotherm model gave a higher  $R^2$  value of 0.93, 0.89 and 0.986 for copper, nickel and lead, respectively, over that of Freundlich model during the adsorption process of the three metals in matrix solution.

**Keywords:** Adsorption; Carbon nanotubes; Granular activated carbon; Isotherm

## INTRODUCTION

Industrialization, uncontrolled use and exploitation of natural resources of the past decades have resulted in increased pollution of the Earth (Bansal and Goyal, 2005). Pollution of the environment by heavy metals is of great concern to governments and environmentalists due to their detrimental effect on a variety of living species (Issabayeva *et al.*, 2007). Pollution of the environment in Malaysia by heavy metals is mainly by electroplating and metal treatment / fabrication industries located in the west coast of the peninsular Malaysia (DOE, 1979; Issabayeva *et al.*, 2007). Research on finding more efficient technology for wastewater treatment to meet prevalent safe standards is constantly receiving the attention of environmental scientists around the world. Previous wastewater treatment efforts had led to the development of various treatment technological options which involved the application of unit operations or unit processes such as chemical precipitation, coagulation, adsorption, ion exchange

and membrane filtration (Georg and Max, 2008). Furthermore, among aforementioned treatment technologies, adsorption had been reported as the most technically and economically viable option (Onundi *et al.*, 2010). Furthermore, research in wastewater treatment by adsorption has resulted in development of different materials for removal of metals from solutions, these materials include: natural product (Nouri *et al.*, 2009; 2011), activated carbon (Issabayeva *et al.*, 2006; Onundi *et al.*, 2010), zeolites, aluminosilicate (Samuel and Osman, 1987), peat kaolin and clay (Chantawong *et al.*, 2003) and polysaccharides (Bong *et al.*, 2004). Recently, Carbon nanomaterials (CNMs) mainly in the form of Carbon nanotubes (CNTs) and Carbon nanofibers (CNFs) are being used as new adsorbents with superior performance due to their high specific surface area and high aspect ratio. Work on the effect of morphology, surface functional groups on adsorption capacity of heavy metals by CNMs had been carried out (Nora and Mamadou, 2005; Kabbashi *et al.*, 2009; Atafar *et al.*, 2010). Work of Li *et al.* (2003) reported Multiwall carbon nanotubes (MWCNTs) as having metal-ion

✉ \*Corresponding Author Email: [mamun@iiu.edu.my](mailto:mamun@iiu.edu.my)  
Tel: +603 6196 4440, Fax: +603 6196 4442

sorption capacity of 3–4 times larger than the widely used powder and Granular activated carbon (GAC). Furthermore, CNTs with rougher surfaces were observed as having improved metal ions adsorption capacity compared to smooth surface ones (Li *et al.*, 2007). However, handling and separation of these CNMs materials from the filtrate were noted as posing a challenge in the application of nanomaterials in water treatment (Nora and Mamadou, 2005). Success had been made in the synthesis of composite nanoscale carbon materials on a macroscale substrate (Vieira *et al.*, 2004; Pingle and Lefferts, 2006; Zhang *et al.*, 2008; Zhou *et al.*, 2009) which could reduce the challenge with the handling of these nano-size materials, however application of these composite materials in metal removal from solution is yet to be extensively investigated. This work explores the possibilities of synthesizing CNT on GAC to form a composite material CNT-GAC and studying the adsorption mechanism of the nano-size composite adsorbent in the removal of copper, nickel and lead ions from aqueous solution. Langmuir and Freundlich isotherm models were employed to describe the adsorption process and the effects of adsorbate pH variation, contact time and adsorbent dosage on metal removal were investigated. The production of CNT-GAC composite adsorbent and effect of pH, contact time and adsorbent dosage on metals adsorption from aqueous solution are reported in this paper. This work was done in 2010, at Bio-environmental Engineering Research Unit (BERU) of the International Islamic University Malaysia (IIUM).

## MATERIALS AND METHODS

Industrial wastewater samples from a semiconductor company (located at Kuala Langat, Malaysia) were collected, preserved and tested for metals content following standard methods (APHA *et al.*, 2005) procedures. The concentration of heavy metals, namely lead, nickel and copper in the industrial wastewater served as the bases for the adsorption study in the laboratory.

### *Adsorbate*

Metals solutions of copper, lead and nickel ions of 1000 mg/L in nitric acid were purchased from Fisher Scientific UK. These were used to produce aqueous solutions of the three metals in a mixed solution, based on the 5-day average metal concentration from samples of actual industrial effluent mentioned above.

### *Adsorbent*

Palm kernel shell based GAC used as substrate material for the CNT production was bought from KDTechology Malaysia. The synthesis of CNT on the GAC was carried out in stages. The first stage involved impregnation of nickel onto GAC using 1 % (w/w) nickel catalyst [ $\text{Ni}(\text{NO}_3)_2$ ]. The nickel catalyst served as seed to nucleate the growth of CNT. The impregnated GAC was then dried overnight in a sonicator water bath at 56 °C. The dry mixture was placed in a thermal Chemical vapour deposition (CVD) reactor for calcination of the mixture by passing  $\text{N}_2$  gas at a flow rate of 300 mL/min under 300 °C for one hour to convert the nickel salt into corresponding nickel oxide. The nickel catalyst attached to the surface of the GAC was then reduced to nickel at 300 °C for another hour in presence of  $\text{H}_2$  gas at a flow rate of 300 mL/min. The final stage of synthesis was carried out in the same tubular CVD reactor at atmospheric pressure by passing carbon source  $\text{C}_2\text{H}_2$  (acetylene gas) at a flow rate of 50 mL/min in conjunction with the hydrogen gas for one hour at 750 °C. The novel CNT-GAC adsorbent was cooled to room temperature in the presence of  $\text{N}_2$  gas in the reactor, before washing it with distilled water to remove fines. The wet CNT-GAC was then dried at 110 °C for two hours and subsequently used for absorption studies at ambient room temperature of 27 °C ( $\pm 2$ ).

### *Adsorbent characterization*

Field emission scanning electron microscope (FESEM, brand Jeoul Jsm-6700F) was employed to study the morphology of the CNT-GAC material, while transmission electron microscope (TEM, brand Jeoul Jem-2010) was used to check the inner structure of the CNT-GAC. Samples of CNT-GAC for Fourier transform infrared (FTIR) analysis were prepared by diluting the adsorbent to 5 % in KBr and cast in disks for analysis to obtain the spectrum from a PerkinElmer precisely of model 100, same procedure was followed by Zvinowanda *et al.* (2009). Specific surface area of the CNT-GAC was measured by Coulter sorptometer machine of model SA-3100, using  $\text{N}_2$  as adsorbent at the liquid nitrogen temperature of -196 °C.

### *Adsorption study*

For each experiment, 50 mL of synthetic water sample containing mixed metals of 2.0 mg/L Cu, 1.5 mg/L Pb and 0.8 mg/L Ni, was prepared based on

industrial wastewater metal concentration and added to a calculated amount of adsorbent in 100 mL shake flask. The pH adjustment of solution was done using 1.0 M HCL and 1.0 M NaOH. The adsorbent in solution was agitated in a mechanical shaker at a speed of 100 rpm at 27 °C ( $\pm 2$ ). Blank solutions were treated similarly without the adsorbent and under control condition as suggested by [Goel et al. \(2005\)](#). The solution was filtered using a Whatman® 0.45  $\mu\text{m}$  filter paper. The results were analyzed for the residual concentration of metals in the filtrate by Atomic adsorption spectrophotometer (ASS) model HGA900.

Equilibrium concentration of metals at different adsorbent dosage (1, 2, 3 and 4 g/L) at ambient temperature was used for the isotherm study. Isotherm studies at equilibrium constant concentration of metals was investigated using the two most widely used model equations of Freundlich ([Freundlich and Hatfield, 1926](#)) and Langmuir ([Langmuir, 1918](#)).

## RESULTS AND DISCUSSION

The macro size composite CNT-GAC was easier to handle in the laboratory than the pure CNT. It was observed that the filtrate after adsorption experiment was clear, having no visible CNT-GAC remaining in solution. This indicates that the macro size composite CNT-GAC retained the CNT on the GAC surface after adsorption, reducing membrane fouling, solving the separation problems earlier recorded in literature and also eliminating the need to centrifuge the filtrate before using the AAS machine.

### Physical and chemical characterization of the adsorbent

The surface of the substrate GAC before the growth of CNT was largely porous as shown in [Fig. 1](#). The morphology of the CNT-GAC as observed by FESEM machine in [Fig. 2](#) shows that the structures were twisted with a rough surface, having average outer diameter of 50 nm.

The TEM images as shown in [Fig. 3](#) and [4](#) revealed the morphology of the CNT-GAC as MWCNT with average internal hollow diameter of 30 nm, with a capped end. This observed structure of CNT on the GAC is inline with the observation reported by [Shoushan et al. \(1999\)](#), [Chai-chih et al. \(2008\)](#) and [Zhang et al. \(2008\)](#) when CNT was produced at same temperature range. Other properties of the substrate GAC and the produced CNT-GAC are shown in [Table 1](#).

The FTIR analysis of CNT-GAC is shown in [Fig. 5](#) and a summary of the peaks and their assignments are given in [Table 2](#). As can be inferred from the FTIR analysis, the acidic functional groups were: carboxyl, carbonyl, lactones and sulphur groups. Generally, these acidic groups on carbon surface produce cation exchange properties ([Goel et al., 2005](#); [Edwin, 2008](#)) and these had been reported to be responsible for higher adsorption of metal ions on carbon surface. Specifically sulphur groups were reported by [Goel et al. \(2005\)](#) as aiding lead ions adsorption. In solution, the dissociation of carboxylic surface groups facilitates the dispersion of the carbon in water by

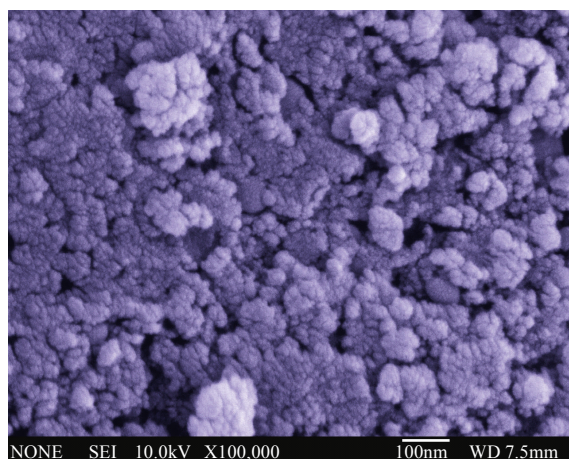


Fig. 1: FESEM Image of GAC surface

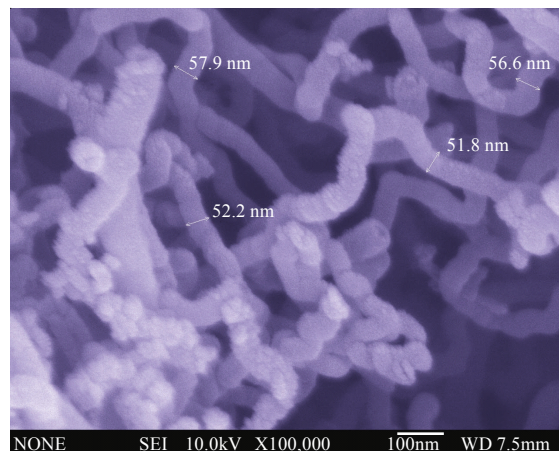


Fig. 2: FESEM Image of CNT grown on GAC surface

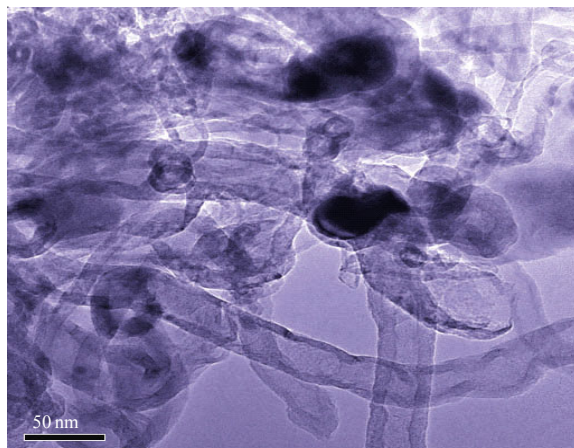


Fig. 3: TEM image of the CNT grown on GAC

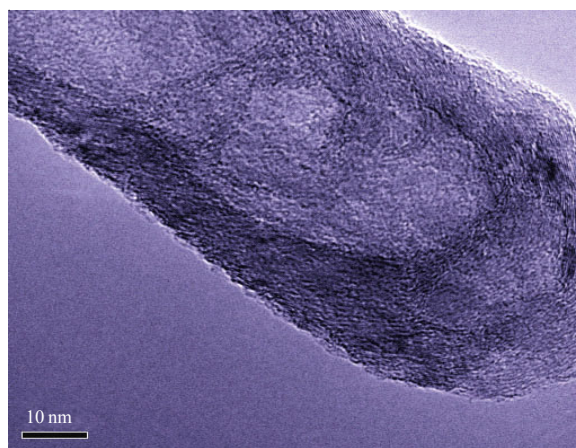


Fig. 4: End cap of the multi-walled CNT on GAC

creating a negative surface charge (Chen *et al.*, 1996). Chen and Wang (2002) emphasized that higher content of carboxylic functional groups could significantly improve adsorption of metal due to the high affinity between them.

#### Effect of pH on adsorption

The pH of the solution had been reported to be important factor in adsorption processes (Corapcioglu and Huang, 1987). The variations in adsorption capacity of CNT-GAC with increasing pH is shown in Fig. 6, it could be inferred that the adsorption capacity of CNT-GAC increased as solution pH increased from pH 3 to 5 and capacity decreases above pH 5.

The mechanism of metals adsorption by the adsorbent CNT-GAC could be explained as follows: the metals (copper, lead and nickel) ions exist predominately as  $M(II)$  ions in highly acidic pH range of 2 to 3. The minimum adsorption observed at low pH could be related to the higher concentration and mobility of  $H^+$  ions, which is favored by high solubility and ionization of metallic salts in acid medium. Thus suggesting that at lower pH value, the surface of the adsorbent is surrounded by hydronium ions, thereby preventing metals ions from approaching the binding sites of the adsorbents. Thus, at higher  $H^+$  concentration, the adsorbents' surface becomes more positively charged such that the attraction between adsorbents and metal cations is reduced. In contrast, as the pH increases, more negatively charged surface becomes available thus facilitating greater metals removal. Thus, the sorption of metal cations increased with increasing pH as the metal ionic species become

less stable in the solution. The maximum adsorption observed in the pH 5 and the subsequent reduction in adsorption capacity was probably due the partial hydrolysis of metal ions, resulting in the formation of  $M(OH)^+$  ions and  $M(OH)_2$ , which would be adsorbed to a greater extent on a less-polar carbon surface of the adsorbents compared to  $M^{2+}$  ions (Onundi *et al.*, 2010). Furthermore, the low solubility of hydrolyzed metals species would have resulted into precipitation of metals at pH above 5, thereby reducing adsorption capacity of CNT-GAC.

#### Effect of contact time

The result of the effect of contact time on percentage metal removal at pH 5 is represented in Fig. 7. The adsorption rate was observed as rapid in the first 5 min, followed by a gradual decrease with time until equilibrium adsorption was noticed at 60 min. The fast adsorption at the initial stage was probably due to the initial concentration gradient between the adsorbate in solution and the number of vacant sites available on the CNT-GAC surface. The progressive decrease in adsorption rate and consequently the attainment of equilibrium adsorption might have been due to reduction in the available active adsorption sites on the adsorbent with time resulting to limited mass transfer of the adsorbate molecules from the bulk liquid to the external surface of CNT-GAC. It was noted that the sorption capacities corresponding to equilibrium varied for each metal, with lead ions recording 99 %, copper 61 % and nickel 20 %, giving metal affinity trend of  $Pb^{2+} > Cu^{2+} > Ni^{2+}$  on the CNT-GAC. The observed trend was in agreement to the findings of Goel *et al.*

Table 1: Properties of substrate GAC and novel CNT-GAC

Parameters	GAC	CNM
Raw material	Palm kernel shell	Palm kernel shell and CNT
Particle size (mm)	1.68 - 2.38	1.68 - 2.38
Ash content	4% max	5% max
Density (g/cm <sup>3</sup> )	0.864	0.784
Surface area (m <sup>2</sup> /g) (BET)	513.3	300.1
Average pore size (Å)	48.5	52.34
Total pore volume (cc/g)	0.622	0.393
Elemental analysis (%)		
C	64.45	57.60
N	23.09	26.22
O	00.00	0.44
Others	32.09	68.18

Table 2: Peaks from CNT spectrum by FTIR

CNT-GAC band position (1/cm)	Possible assignments
3448	O-H stretching (intermolecular diametric)
3200	C-H stretching (alkenes) carboxyl
2045	C≡N stretching (alkynes)
1697	C=O Stretching (aldehydic) carbonyl
647	S-O Bends (sulfates)

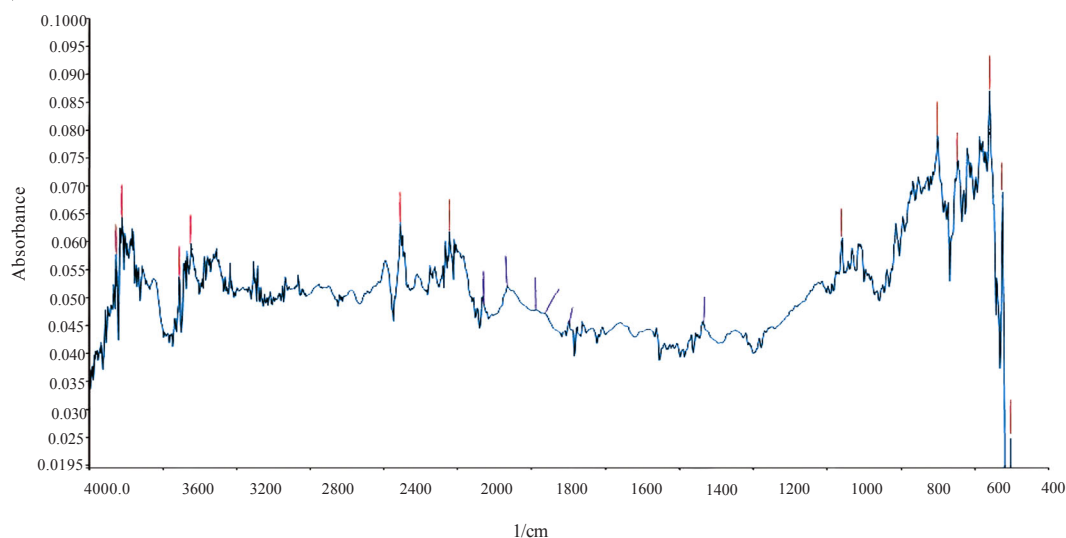


Fig. 5: FTIR Spectrum of CNT-GAC

(2005), who suggested that the presence of sulphur functional group on adsorbent favours lead ions selective sorption.

#### Effect of adsorbent dosage

The result of variation of adsorbent capacity with increment in dosage using a contact time of 60 min. at pH 5 is shown in Fig. 8. It was observed that as the dosage increased from 0.5 to 1 g/L the metals adsorption capacity increased from 0.119 mg/g to 0.853 mg/g for lead ions, while capacity for copper ions increased from 0.109 mg/g to 1.013 mg/g. A decline in capacity was noticed beyond 1 g/L dosage for copper and lead ions; however the capacity for nickel ions increased from 0.01

mg/g at 1 g/L dosage to a maximum of 0.07 mg/g at 2 g/L. This implies that optimum dosage lies around 1 g/L to 2 g/L, therefore further increment in dosage does not result to a significant increment in adsorption capacity.

The initial increment in adsorption capacity with increase in adsorbent dosage was expected because as the adsorbent dose increases the number of adsorbent particles increases, thus more surface areas were available for metals attachment, similar trend was reported by Acharya (2009). It is plausible to suggest that with higher dosage of adsorbent there would be greater availability of exchangeable sites for metal ions (Acharya, 2009). As noticed in Fig. 8, further increment in adsorbent dosage beyond maximum adsorption capacity (at 1g/L for Cu<sup>2+</sup>

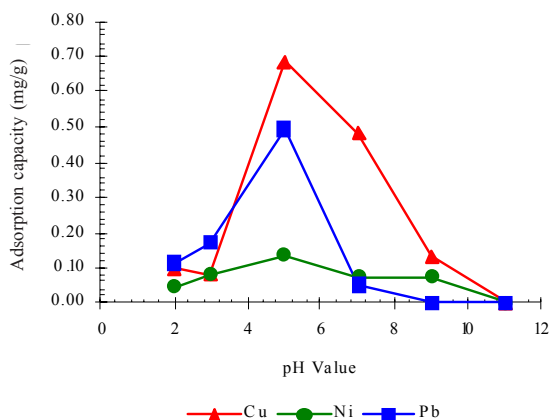


Fig. 6: Effect of pH on adsorption of metals onto CNT-GAC from a mixed solution (adsorbent dosage = 0.5 g/L, agitation = 100 rpm and time = 60 min)

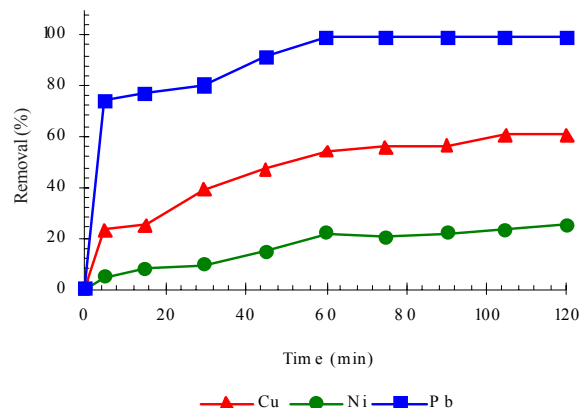


Fig. 7: Effect of contact time on adsorption of Cu(II), Ni(II) and Pb(II) (constant temperature = 27 (±2)°C, pH=4 and adsorbent dose = 4 g/L)

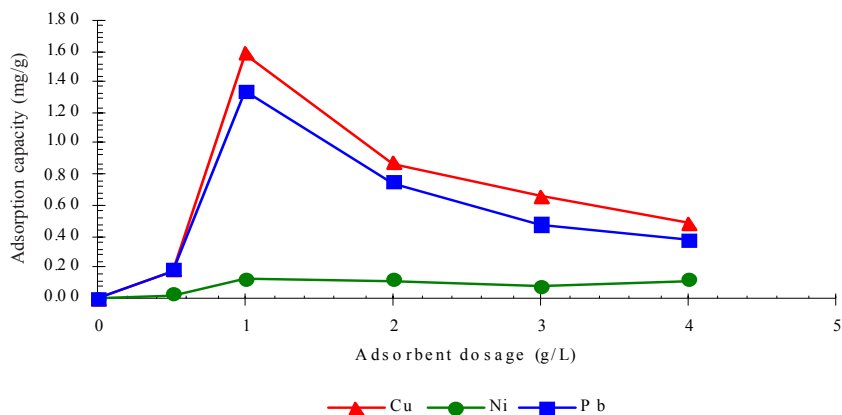


Fig. 8: Effect of adsorbent dosage (g/L) on metals adsorption capacity (in 50 mL solution at pH5, agitation period = 60 min, agitation speed at = 100 rpm, metals having different initial concentration)

Table 3: Isotherm parameters for metals adsorption at 27(±2) °C and pH 5

Metals	Equation	Constants		$C_s(\text{equation})$	$R^2$
Copper	Langmuir	$a=0.889$	$b=3.5961$	$C_s = \frac{x}{m} = \frac{(0.8892)(3.5961)}{1 + 3.5961C_e}$	0.928
	Freundlich	$K_f=-0.098$	$n=1.9305$	$C_s = \frac{x}{m} = 0.09785 \cdot C_e^{1/1.9305}$	0.850
Nickel	Langmuir	$a=-0.069$	$b=-0.8656$	$C_s = \frac{x}{m} = \frac{(-0.06927)(-0.86564)}{1 - 0.86564C_e}$	0.889
	Freundlich	$K_f=-0.536$	$n=0.3825$	$C_s = \frac{x}{m} = 0.5365 \cdot C_e^{1/0.3825}$	0.726
Lead	Langmuir	$a=0.682$	$b=86.2609$	$C_s = \frac{x}{m} = \frac{(0.6824)(86.2609)}{1 + 86.2609C_e}$	0.986
	Freundlich	$K_f=-0.052$	$n=14.1581$	$C_s = \frac{x}{m} = -0.05166 \cdot C_e^{1/14.1581}$	0.905

and  $\text{Pb}^{2+}$ , 2 g/L for  $\text{Ni}^{2+}$ ) caused a decline. This reduction in capacity could be due to the overlapping of the adsorption sites as a result of overcrowding of adsorbent particles when the adsorbent dosage is increased above 2 g/L. This imposed a screening effect on the dense outer layer of the adsorbent, thereby shielding the binding sites from metals as explained by Garg *et al.* (2003) and Babel and Kurniawan (2004).

#### Equilibrium adsorption isotherms

The equilibrium adsorption data was fitted into the Langmuir and Freundlich equations to yield the model parameters with correlation coefficients ( $R^2$ ) shown in Table 3. It was observed that results fitted better in the Langmuir model in terms of  $R^2$  value, recording 0.99 for lead, 0.93 for copper and 0.89 for nickel. The trend of affinity for metals by the CNT-GAC surface observed in terms of constant "a" was  $\text{Cu}^{2+} > \text{Pb}^{2+} > \text{Ni}^{2+}$ . This could have been due to the initial concentration of metals following same trend. While the affinity of the metals for the adsorbent surface in terms of value "b" in the Table 3 varied in the trend  $\text{Pb}^{2+} > \text{Cu}^{2+} > \text{Ni}^{2+}$ , this was probably due to the acidic functional groups present on the CNT-GAC surface. Particularly the sulphate group is assumed to favour lead ions adsorption over other metals, while the other acidic group generally favours metals adsorption.

#### CONCLUSION

A novel CNT-GAC composite was successfully produced which was easier to handle compared to pure nanomaterial alone. The thermal CVD process reported here was a cleaner method compared to floating catalyst CVD process for CNT production, as the CNT were synthesized on relatively large substrate GAC. Experimental results on adsorption conditions indicated that copper, nickel and lead ions have highest adsorption capacity at pH 5, while the maximum adsorbent dosage was 1 g/L for copper and lead ions recording a sorption capacity of 1.013 and 0.853 mg/g respectively, and 2 g/L for nickel recording 0.07 mg/g. Langmuir isotherm adsorption model having higher  $R^2$  value of 0.93, 0.89 and 0.99 for copper, nickel and lead ions, respectively, described the adsorption process better than Freundlich model for the three metals. This novel material opens new door for various usage of the nanomaterials in different fields of application in the chemical, petrochemical industries and wastewater treatment plants, though more work is needed for the mass production of the material at reduced cost.

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#### AUTHOR (S) BIOSKETCHES

**Onundi, Y. B.**, M.Sc., Department of Biotechnology Engineering, Faculty of Engineering, International Islamic University Malaysia (IIUM), Jalan Gombak, 53100 Kuala Lumpur, Malaysia, Kuala Lumpur, Malaysia. Email: [onudy1759@gmail.com](mailto:onudy1759@gmail.com)

**Mamun, A. A.**, Ph.D., Department of Biotechnology Engineering, Faculty of Engineering, International Islamic University Malaysia (IIUM), Jalan Gombak, 53100 Kuala Lumpur, Malaysia, Kuala Lumpur, Malaysia. Email: [mamun@iium.edu.my](mailto:mamun@iium.edu.my)

**Al Khatib, M. F.**, Ph.D., Department of Biotechnology Engineering, Faculty of Engineering, International Islamic University Malaysia (IIUM), Jalan Gombak, 53100 Kuala Lumpur, Malaysia, Kuala Lumpur, Malaysia. Email: [maan@iium.edu.my](mailto:maan@iium.edu.my)

**AlSaadi, M. A.**, M.Sc., Department of Biotechnology Engineering, Faculty of Engineering, International Islamic University Malaysia (IIUM), Jalan Gombak, 53100 Kuala Lumpur, Malaysia, Kuala Lumpur, Malaysia. Email: [mdsd68@yahoo.com](mailto:mdsd68@yahoo.com)

**Suleyman, A. M.**, Ph.D., Department of Biotechnology Engineering, Faculty of Engineering, International Islamic University Malaysia (IIUM), Jalan Gombak, 53100 Kuala Lumpur, Malaysia, Kuala Lumpur, Malaysia. Email: [suleyman@iium.edu.my](mailto:suleyman@iium.edu.my)

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