REMOVAL OF CADMIUM IONS FROM SYNTHETIC WASTEWATER BY USING
Pennisetum purpureum (ELEPHANT GRASS) AS LOW COST
BIODEGRADABLE ADSORBENT (BIOSORBENT)

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Abstract: At present, heavy metal pollution is a major environmental concern and the adsorption technique is a potent method for removal of these heavy metals from wastewater. Activated carbon is one of the best adsorbents for metal ions removal but it is sometimes restricted due to high cost and problems with regeneration hamper large scale application. Low cost adsorbent is alternatively being introduced to replace activated carbon since it is available in large quantity, renewable and inexpensive. Hence, *Pennisetum purpureum* (elephant grass) was investigated for its potential in cadmium ions removal. The adsorbent was characterized by Fourier Transforms Infrared Spectroscopy (FT-IR), Scanning Electron Microscopy (SEM), Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) analyses. The effects of pH (1 to 5), initial metal ion concentration (5 to 25 mg/L), contact time (10 to 60 minutes) and adsorbent dosage (0.2 to 1.0 g) on cadmium ions removal were conducted by batch adsorption experiments. In this study, the FT-IR results demonstrated that the functional groups for untreated and nitric acid-treated *P. purpureum* mainly consisted of carbonyl, carboxyl, hydroxyl and amine groups which are able to bind with positively charged cadmium ions. SEM micrographs have proven that nitric acid modification would remove the surface impurities of *P. purpureum*, which increased the surface roughness, produced deep, open pores and better pore size distribution. From the BET and BJH analyses, the treated *P. purpureum* was mesoporous, had larger surface area and pore volume compared to untreated *P. purpureum*. The best pH, adsorbent dosage and contact time were pH 4, 0.6 g and 30 minutes, respectively. The highest removal percentage of cadmium ions for both untreated and treated *P. purpureum* were 92% and 98% correspondingly. The results shown strengthened the fact that both biosorbents have great potential in cadmium ions removal.

Keywords: Undergraduate research, adsorption, low cost adsorbent, cadmium, *Pennisetum purpureum*

Introduction

The presence of heavy metals in wastewater is a major hazard to surface water, animals and human health. Heavy metals are produced by electroplating, dyeing, metal finishing and chemical processing industries. Moreover, heavy metals are non-biodegradable and highly soluble in aquatic environment. They can be absorbed by living organisms and may bio-accumulate in the human body if they enter the food chain. Heavy metals can cause serious health problems if consumed beyond the permitted concentration (Babel & Kurniawan, 2004). Therefore, it is necessary to treat metal-contaminated wastewater prior to its discharge to the environment.

One of the non-essential metals is Cadmium (Cd), which is poisonous for plants, humans and animals (Raja *et al*., 2009). It is a soft, silver-white metal which does not have a defined taste or odor. Location of the periodic table is in group IIB. Atomic number is 48 and atomic mass is 112.411. The most severe disease caused by cadmium poisoning is "*Itai-Itai*" disease in Japan. Cd toxicity is associated with pulmonary, renal, hepatic, skeletal, reproductive and cardiovascular dysfunctions (Hong *et al*., 2004; Koyu *et al*., 2006; Tellez-Plaza *et al*., 2008). It is also classified as a group I human carcinogen by the International Agency for Research on Cancer (IARC, 1993). Conventional treatments for cadmium ions removal such as chemical precipitation, ion exchange, oxidation and reduction, filtration and membrane separation have significant disadvantages such as incomplete removal, high-energy requirements, and production of toxic sludge (Barakat, 2011). Of all the methods, adsorption has been found to be effective and low cost (Venkatesan & Senthilnathan, 2013). Although commercial activated carbon is widely used for the removal of heavy metals, it is sometimes restricted for industrial use due to its high cost and industries following different strategies on disposal of end-of-life sorbent. This happens mainly in developing countries and under developing countries. Hence, there is

a need to derive a low cost adsorbent for the removal of cadmium from wastewater. Thus, in this study *P. purpureum* was selected for treating cadmium in synthetic wastewater in order to explore its potential as a low cost biosorbent at its promising characteristics and performance in control adsorption batch experiments.

Materials and Methods
Biomass Pre-treatment

P. purpureum was collected from around Gong Badak, Kuala Nerus, Terengganu. The raw P. purpureum was washed with distilled water to remove all the impurities. The sample was bottomed with aluminum foil and put into universal oven for drying purpose at 105˚C for 24 hours until a constant mass was achieved. The dried *P. purpureum* was cut into pieces of approximately 0.5 cm (Babarinde & Babalola, 2010). Zip lock bags were prepared to store the untreated and treated biosorbents, labeled clearly and placed into desiccator for future use. Similar procedure was followed for the preparation of treated *Pennisetum purpureum*. 10g of the untreated *P. purpureum* was immersed into 100mL of 2M nitric acid (HNO_3) solution, agitated in a water bath shaker at 200rpm for 6 hours at room temperature (Raoul *et al*., 2016). The acid solution was filtered off, washed with distilled water and soaked in 0.1M sodium hydroxide (NaOH) to remove residual acid. The solution was filtered off and washed with distilled water again, then oven-dried at 105°C for 24 hours. Both biosorbents were prepared and kept dry until the time of usage.

Reagents and Equipment

All the chemical reagents used in this study are analytical grade. 1000 mg/L of cadmium nitrate tetrahydrate ($Cd(NO_3)_2 \cdot 4H_2O$) stock solution was prepared by dissolving requisite amount of powder in 1 litre of deionized water. The stock solution was diluted to required concentrations, followed by pH adjustment with 0.1M HNO_3 and 0.1M NaOH, measured by pH meter. The spectrophotometric determination of cadmium ion was done by adding 1 mL of sample with adjusted pH to the range of pH 5.5 to pH 6.1, followed by 1 mL of 1.39×10^{-1} M Alizarin red S ($C_{14}H_8O_7S$) in a 10 mL measuring cylinder. Deionized water was added to mark and mixed well. This is because Alizarin red S reacts in slightly acidic solution with cadmium to give a deep greenish yellow

chelate which has a maximum absorption at 422 nm (Ullah & Haque, 2010). UV-Vis spectrophotometer was used for residual metal ions analysis after each biosorption process at 422 nm.

For the characterization of both biosorbents, Fourier Transform Infrared Spectroscopy (FT-IR) was used to determine the surface functional groups for both untreated and treated *P. purpureum* which are responsible for bonding between the *P. purpureum* and cadmium ions. The spectral were recorded ranging from 4000-400 cm^{-1}. Scanning Electron Microscopy (SEM) was used to characterize the surface morphology and fundamental physical properties of the adsorbent materials. The analysis was conducted at 1000 times magnification with shown SEM micrograph. The specific surface area and pore volume were determined by using nitrogen adsorption-desorption isotherm properties. The analysis was carried out by using Micromeritics ASAP 2000, available in Central Laboratory, UMT.

Batch Biosorption Procedure

The biosorption was carried out by using batch method to investigate the effect of pH, contact time, initial metal ion concentration and adsorbent dosage on the biosorption of cadmium ions. For the fixed variables at the beginning of the experiment, 0.5g biosorbent was contacted with 25mL of 20 mg/L cadmium solution in a sealed 100mL conical flask and agitated vigorously by using water bath shaker. The shake speed was kept constant at 250 rpm for 30 minutes. The variables could be changed based on the best condition obtained. The pH of the solution was adjusted with 1M HCl and 1M NaOH in the range of 1 to 5. The sealed flasks were agitated for contact time intervals of 10 minutes to 60 minutes. The influence of initial metal ion concentration (5 to 25 mg/L) and adsorbent dosage (0.2 g to 1.0 g) was also investigated in this study. Samples were collected from the flasks and the residual cadmium ion concentration in each flask was analyzed. The suspensions were filtered through Whatman filter paper and the supernatants were analyzed by measuring absorbance at maximum wavelength of 422nm using UV-Vis Spectrophotometer. The biosorption experiments were repeated in triplicate to reduce any possible random errors. The percentages of removal of cadmium ions by both biosorbents were calculated using Equation (1)

$$\text{Percentage of removal (\%)} = \frac{c_o - c}{c_o} \times 100\% \qquad (1)$$

Where
c_o = Initial cadmium ions concentration, mg/L
C = Final concentration of the solution after experiment, mg/L

Results and Discussion
Fourier Transform Infrared Spectrocopy (FT-IR)

FT-IR spectra analysis was used mainly to identify the existence of available functional groups (carboxyl, hydroxyl and amine groups) that were capable of adsorbing cadmium ions (Qiao *et al*., 2014). FT-IR spectra of untreated and treated *P. purpureum* are shown in Figure 1 and Figure 2 respectively. The peaks were observed at 3375.64 cm^{-1} and 3275.67 cm^{-1} in untreated and treated biosorbents respectively, which indicated the presence of intermolecular hydrogen bonding with functional groups of O-H stretching and N-H stretching (Kamarullah *et al*., 2015). The FT-IR peaks were also observed at 1644.08 cm^{-1} and 1656.73 cm^{-1} in untreated and treated biosorbent accordingly. At this range, the C=C stretching and C=N stretching from alkenes and imine or oxime group were identified. However, the C=C bending of untreated biosorbent was identified at wave number of 803.94 cm^{-1}. Besides that, C-O stretch and C-N stretch were discovered for both untreated and treated biosorbent, at wave number of 1054.28 cm^{-1} and 1055.42 cm^{-1} correspondingly.

Moreover, C-H stretching from alkanes group was identified and sp^3 C-H absorption was observed for both untreated and treated biosorbents. The FT-IR bands at 2894 - 2917 cm^{-1} corresponded to lignin composition. In short, both untreated and treated biosorbents consist of functional groups with lone pairs of electrons which are able to attract and bind with positively charged cadmium ions. It can be suggested that *P. purpureum* is basically composed of lignin, cellulose and hemicelluloses structure, and all these components contribute as active sites for adsorption of metal ions. The treated biosorbent shifted to higher absorption frequency values than untreated biosorbent, which denoted that treated biosorbent has a higher adsorption capacity than untreated biosorbent. The detailed categorization of wave number corresponded to type of vibration and functional group as shown in Table 1. The presence of carboxyl, carbonyl, hydroxyl and amine functional groups on biosorption of cadmium ions mainly contributes to ion exchange, physical adsorption, electrostatic attraction and complexation mechanisms (Javanbakht *et al*., 2014).
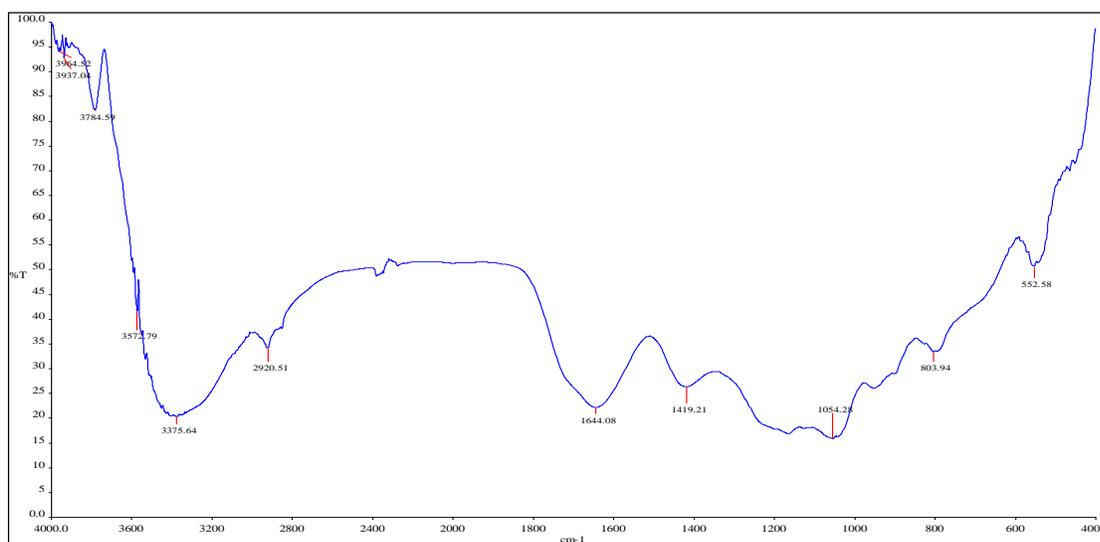

Figure 1: FT-IR spectrum of untreated *P. purpureum.*
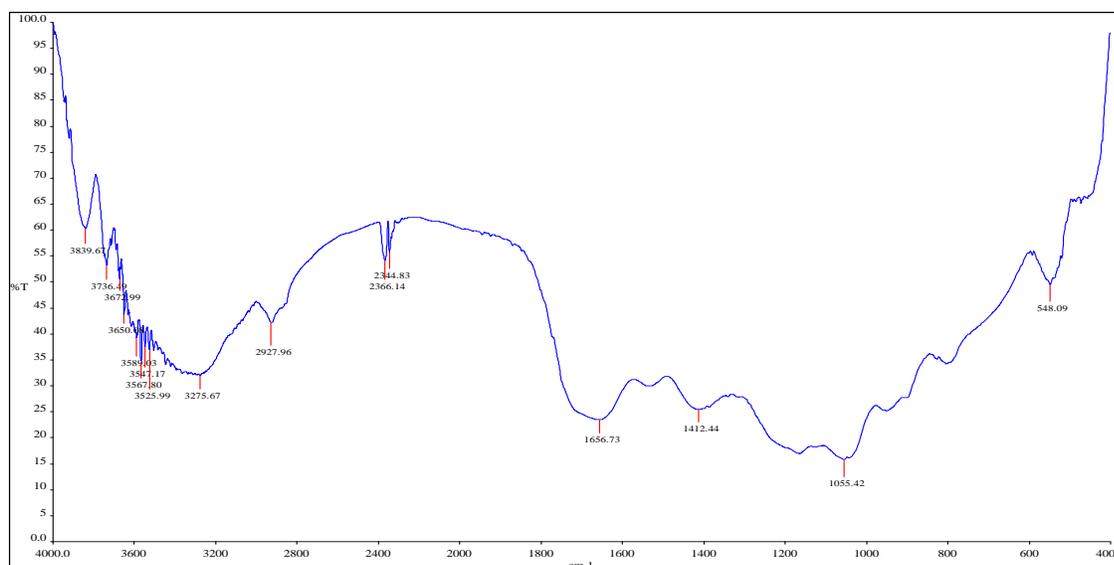
Figure 2: FT-IR spectrum of treated *P. purpureum.*

Table 1: FT-IR spectra comparison between untreated and treated *P. purpureum.*

Untreated *P. purpureum*			Treated *P. purpureum*		
Wavelength (cm^{-1})	Type of vibration	Functional Group	Wavelength (cm^{-1})	Type of vibration	Functional Group
803.94	C=C bending, C-H bending	Alkene, 1,4-disubstituted or 1,2,3,4-tetrasubstituted	1055.42	C-O stretch, C-N stretch, S=O stretch	Primary alcohol, Vinyl ether, Amine, Alkyl aryl ether, Sulfoxide
1054.28	C-O stretch, C-N stretch	Primary alcohol, Vinyl ether, Alkyl aryl ether, Amine	1412.44	O-H bending	Alcohol, Carboxylic acid
1419.21	C-H bending, O-H bending	Alkane, Alcohol, Carboxylic acid	1656.73	C=C stretch, C=N stretch	Alkene, Imine/Oxime
1644.08	C=N stretch	Imine/oxime	2927.96	C-H stretch, O-H stretch, N-H stretch	Alkane, Carboxylic acid, Alcohol, Amine salt
2920.51	-C-H stretch	Alkane	3275.67	O-H stretch, N-H stretch	Alcohol, Carboxylic acid, Aliphatic primary amine
3375.64	N-H stretch	Aliphatic primary amine	3525.99 3547.17 3567.80 3589.03 3650.08 3672.99	O-H stretch	Alcohol
3572.79	O-H stretch	Alcohol			

Scanning Electron Microscope (SEM)

As shown in Figure 3 (a) and (b), the surface of untreated *P. purpureum* was irregular, smooth and compact. The presence of pore and cavities was observed but they posed a close structure. The treatment of *P. purpureum* with HNO_3 showed improvement in physical characteristics as shown in Figure 4 (a) and (b). The surface of treated *P. purpureum* was rougher compared to that untreated. The pores and cavities could be easily observed and the pores were in deeper and wider structure, which indicated that the treated *P. purpureum* had more porous structure and provided a better pore size distribution. These characteristics are fundamental for biosorption process since it makes the active sites more accessible. The grooves on the treated biosorbent were formed by removing the impurities on the surface of adsorbent during the nitric acid treatments, which may help to increase the roughness and binding sites on material surface (Wang *et al*., 2016). The SEM micrographs of HNO_3 treated biosorbent also presented the clear removal of a surface layer and empty space between the biosorbent, which indicated the removal of impurities such as higher hemicellulose layer in natural fibres (Kamarullah *et al*., 2015).


(a)

(b)
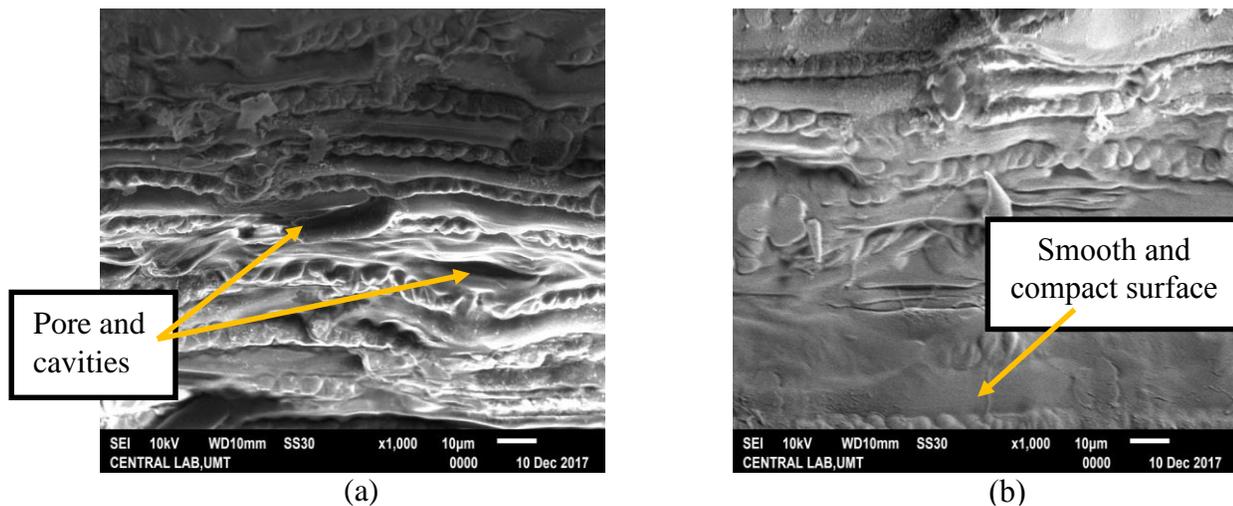
Figure 3: SEM micrographs of untreated *Pennisetum purpureum*.

(a)

(b)
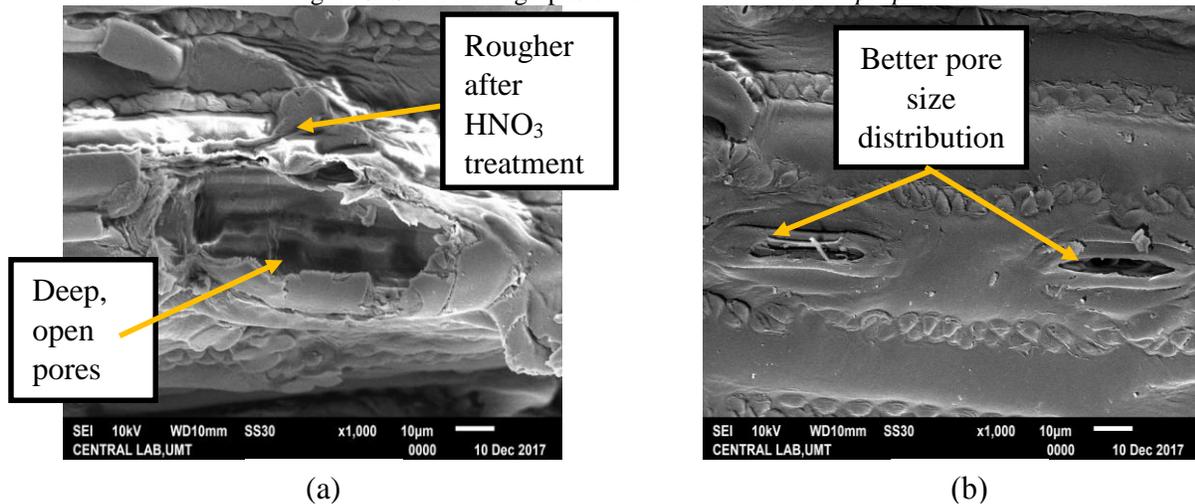
Figure 4: SEM micrographs of treated *Pennisetum purpureum*.

Analysis of Surface Area and Porosity (ASAP)

The texture properties such as surface area and pore volume play a significant role in sorption process of metal ions (Ciesielczyk *et al*., 2016). Surface area and pore size distribution of untreated and treated *P. purpureum* were measured using BET (Brunauer-Emmett-Teller) and BJH (Barrett-Joyner-Halenda) analysis. Table 2 shows the summary of BET and BJH analysis results for untreated and treated *P. purpureum*.

Table 2: BET and BJH analysis results for untreated and treated *P. purpureum*.

Sample	BET surface area (m^2/g)	BJH adsorption cumulative pore volume (cm^3/g)	BJH adsorption average pore diameter (nm)
Untreated *P. purpureum*	1.1077	0.003752	52.6709
Treated *P. purpureum*	1.3385	0.005987	27.7506

From Table 2, BET surface area of *P. purpureum* after nitric acid modification showed an increment from 1.1077 m^2/g to 1.3385 m^2/g, which in turn increased the pore volume of *Pennisetum purpureum*. This indicates that nitric acid modification on biosorbent enhances the porous characteristics such as surface area, pore volume and better pore size distribution. However, nitric acid modification of *P. purpureum* decreased the BJH adsorption average pore diameter from 52.6709 nm (macropores) to 27.7506 nm (mesopores). Zhu *et al.* (2017) suggested using mesoporous materials for heavy metals adsorption due to its exceptionally larger surface area, regular pore structure and tunable pore size. Therefore, all these characteristics help to explain the higher adsorption capacity of nitric acid-treated *P. purpureum* compared to untreated *P. purpureum*.

The Effect of pH

The initial pH of solution is one of the most important parameters which will affect the surface charge of biosorbent and uptake of heavy metals from wastewater (Tumin *et al.*, 2008). Figure 5 shows the graph of percent removal against varing pH. For untreated *P. purpureum*, the uptake of cadmium ions increased from 27.5% to 92.8% when the pH increased from pH 1 to pH 4. Treated *P. purpureum* also displayed an increase in cadmium ions uptake from 34.1% to 97.8% within pH 1 to pH 4. The minimum adsorption value was observed for both biosorbents at pH 1. This is because a highly acidic solution with low pH can cause the net charge of the active sites on the surface of biosorbent to become positive since it is surrounded by hydronium ions (H$^+$) (Wong *et al.*, 2003). Therefore, the reduction in the number of negatively charged sites prevented the adsorption of positively charged cadmium ions on the surface of biosorbent.

In contrast, more negatively charged sites become available when the pH of solution increases. As the pH increases, the functional groups of biosorbent become deprotonated and are more accessible for the attraction of metal ions, thus increase the adsorption of metal cations (Anayurt *et al.*, 2009). Both untreated and treated *P. purpureum* showed a slight decrease in percent removal at pH 5, which were 90.0% and 96.8% respectively. This is due to the occurrence of cadmium precipitation (Tumin *et al.*, 2008). At this stage, the Cd(OH)$^+$ and Cd(OH)$_2$ are present in large quantities compared to Cd^{2+}, which affect the adsorption of Cd^{2+}. Hence, the result demonstrated that the best pH for both biosorbents in adsorption of cadmium ions is pH 4.
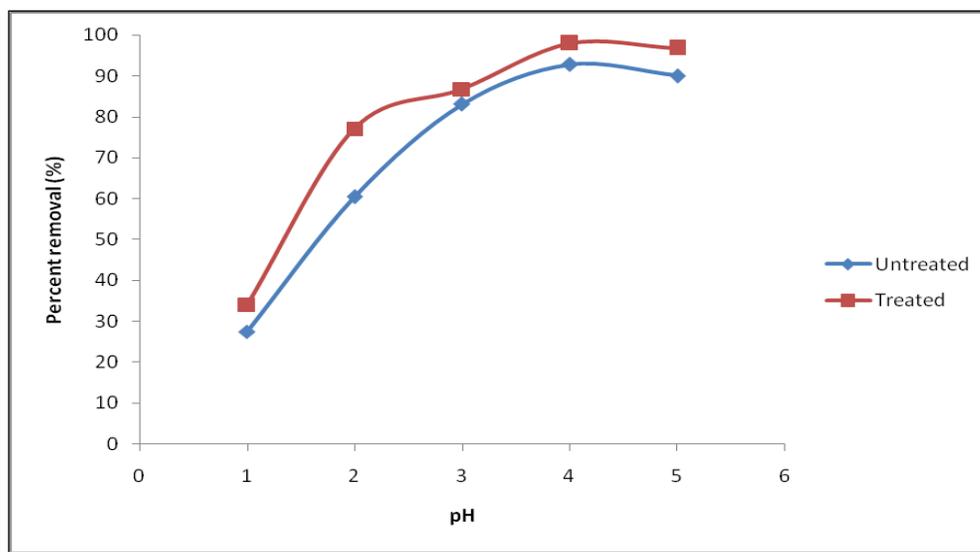

Figure 5: The effect of pH on the adsorption of Cd(II) ions.

The Effect of Initial Metal Ion Concentration

The initial metal ion concentration determines the amount of metal ion biosorbed in the condition when there are available binding sites. Figure 6 shows the result of the effect of initial metal ion concentration on the uptake of cadmium ions. Based on the result obtained, the removal rate increased from 63.3% and 74.8% at 5 mg/L to 92.3% and 97.4% at 25 mg/L for both untreated and treated *P.*

purpureum respectively. This is due to the higher initial concentration that leads to a higher affinity of metal ions towards active sites (Al-Asheh *et al.*, 2003). The higher initial metal ion concentration also indicates the higher availability of metal ions for adsorption. Therefore, the collision between cadmium ions and the biosorbent increases, which in turn increases the rate of reactions, causing the removal rate to increase.
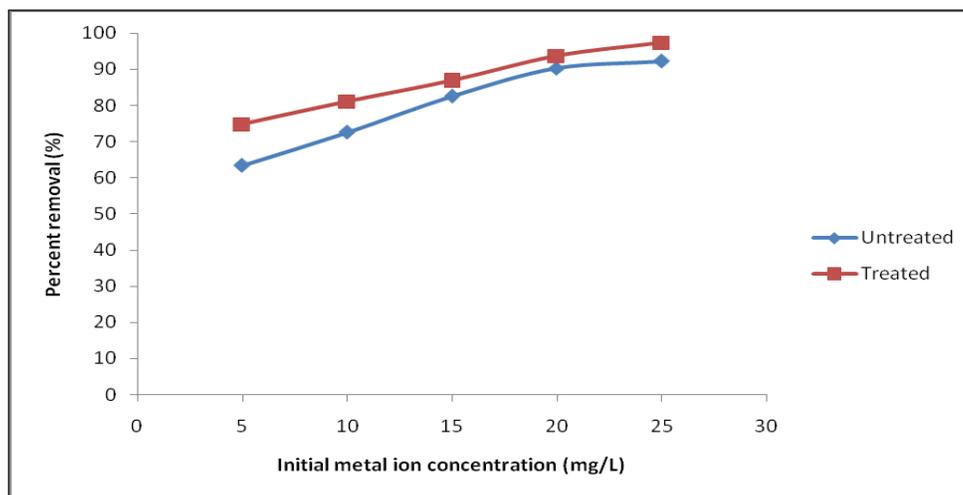

Figure 6: The effect of initial metal ion concentration on the adsorption of Cd(II) ions.

The Effect of Contact Time

Figure 7 displays the effect of contact time onto the biosorption of cadmium ions. The biosorption of both untreated and treated *P. purpureum* increased rapidly within the first 30 minutes. Therefore, 30 minutes was taken as the best contact time. It is important to note that the adsorption of cadmium ions proceeded in 2 stages for both biosorbents. At the first stage which lasted for 30 minutes, the maximum removal rate was 96.3% and 97.0%

for both untreated and treated *P. purpureum* respectively. This can be explained by the availability of all the active sites on the surface of biosorbent (Rao *et al.*, 2010). In the second stage, the removal rate of cadmium ions slightly decreased and achieved equilibrium. This indicates that the rate of biosorption slows down due to the adsorption of cadmium ions that blocks the pores and uses up the available binding sites on the surface of biosorbent (Babarinde & Babalola, 2010).
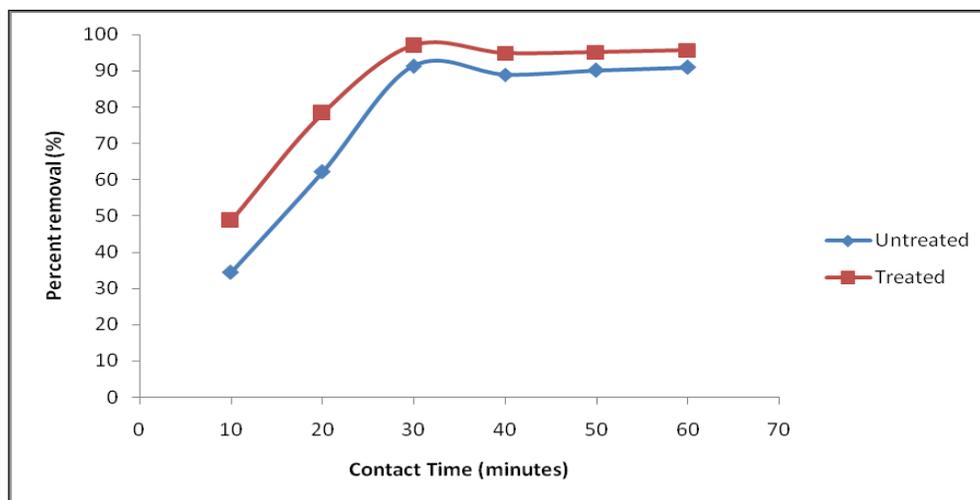
Figure 7: The effect of contact time on the adsorption of Cd (II) ions

The Effect of Adsorbent Dosage

The adsorbent dosage is another important parameter which affects the biosorption of metal cations. Figure 8 shows the effect of adsorbent dosage on the percent removal of cadmium ions from aqueous solution. The trend of the graph demonstrated that the metal uptake increased from 21.3% and 29.8% with 0.2 g of adsorbent up to 86.5% and 97.3% with 0.6 g of adsorbent for both untreated and treated *P. purpureum* respectively. This is due to limited amount of cadmium ions present in aqueous solution exposed to a larger amount of active sites at higher adsorbent dosage, which can achieve a high adsorption percentage at appropriate dosage (Babel & Kurniawan, 2004). Therefore, 0.6 g of adsorbent is considered as the best adsorbent dosage for cadmium ions removal. However, the adsorption percentage decreased sharply with an increase in adsorbent dosage after 0.6 g. This is due to the presence of excessive amount of adsorbent causing the overcrowding of adsorbent particles, and hence overlapping the active sites and make it unavailable for adsorption of cadmium ions (Garg *et al*., 2003). Besides that, high amount of adsorbent dosage would impose a screening effect on the surface of adsorbent, thereby shielding the active sites from the adsorption of metal ions (Tumin *et al*., 2008).
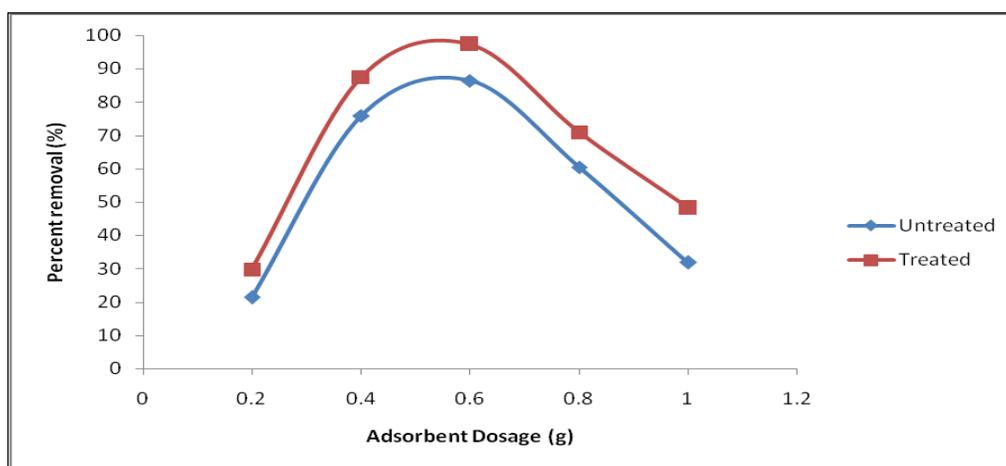

Figure 8: The effect of adsorbent dosage on the adsorption of Cd(II) ions.

Conclusion

This study has investigated the potential of *P. purpureum* as low cost biosorbent for cadmium ions removal. As a conclusion, the derivation of *P. purpureum* as a low cost biosorbent is an ideal way to replace commercial activated carbon for treating metal ions present in wastewater. The FT-IR analysis indicated that the functional groups for both biosorbents consist of carbonyl, carboxyl, hydroxyl and amine groups which are able to bind with the positively charged cadmium ions. The presence of these functional groups mainly contribute to ion exchange, physical

adsorption, electrostatic attraction and complexation mechanisms.

SEM micrographs have shown that untreated *P. purpureum* had irregular, smooth and compact surface. After the nitric acid treatment, it showed an improvement in physical characteristics such as deep, open pores and better pore size distribution by removing the impurities on the surface of biosorbent, which may help to increase the roughness and make the active sites more accessible. The ASAP analysis revealed that the surface area and pore volume of treated *P. purpureum* (1.3385 m^2/g and 0.005987 cm^3/g respectively) were larger than untreated *P. purpureum* (1.1077 m^2/g and 0.003752 cm^3/g respectively). However, nitric acid modification of *P. purpureum* decreased the pore diameter from macropores (52.6709 nm) to mesopores (27.7506 nm). It is suggested that mesoporous materials are preferable for adsorption of heavy metals due to their exceptionally larger surface area, regular pore structure and tunable pore size.

Moreover, the best conditions were obtained from the batch adsorption experiments. For both untreated and treated *Pennisetum purpureum*, the best pH, contact time and adsorbent dosage were determined at pH 4, 30 minutes and 0.6 g, respectively. In addition, the initial metal ion concentration had specific influence on the final removal percentage of Cd (II) ions. It was found that the Cd (II) percent removal was directly proportional to the initial metal ion concentration. The highest Cd (II) ion removal achieved using untreated *P. purpureum* was 92% while the nitric acid-treated *P. purpureum* exhibited 98% removal.

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