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Biosorption of Heavy Metal by Algae Biomass in Surface Water

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Abstract

Discharging wastewater containing heavy metals of Cu, Pb, Zn and Cd into water bodies can cause toxicity in plants and aquatic animals and some of them will be unable to survive except algae. Wastewater treatment method to remove heavy metal contaminants includes chemical precipitation, ion exchange, membrane, filtration, adsorption using activated carbon. However, these methods are either expensive or have other disadvantages such as high energy consumption and inefficiencies when existing heavy metals are at trace concentration. Biosorption using algae biomass can be an alternative method to eliminate heavy metals. The objective of the project is to investigate the capability of Marine Algae (MA) and Freshwater Algae (FA) biomass in adsorbing heavy metals of Cu, Pb, Zn and Cd from water medium using synthetic water and industrial water. MA and FA were obtained from the eastern coast of Pulau Ubin and local fish farm respectively. After being fully washed with deionised water, dried in a furnace for 105˚C, they are grinded to pass 1 mm^2 of siever. MA and FA were characterised using FTIR to determine their functional groups. An industrial water was collected from industrial discharge from metal factories in northern side of Singapore. Effect of adsorption time, adsorbent concentration, and pH were studied. The result showed that FA and MA had a higher capability in adsorbing a total metal of about 40 ppm level from an industrial water, or 4 times than synthetic water concentration, at the same adsorbent dosage of 50 mg. In conclusion, the presence of various functional groups, hydroxyl, carboxylic and amine groups, in all MA and FA samples had enabled the algae biomass to adsorb heavy metals of Cu, Pb, Cd and Zn from synthetic and industrial water. Due to their biosorptive properties and fast adsorption capability, algae could be a potential method for cleaning up surface water or post-treatment of wastewater and minimise the cost of eutrophication.

Keywords

Algae Biomass, Biosorption, Eutrophication, Heavy Metal, Surface Water,

Wastewater

1. Introduction

Many countries have undergone rapid urbanisation and industrialisation over the last few decades. As a consequence, urban stormwater runoff increases directly with imperviousness and the degree of watershed development. Surface waters are forced to accommodate larger volumes of stormwater runoff that return on a more frequent basis. The change in watershed hydrology associated with urban development also causes channel widening and scour, and the introduction of larger amounts of suspended and dissolved sediments, such as heavy metals into river streams.

Heavy metals are toxic and will not only cause the pollution of water bodies but also potential public health problems and risks [1]. Hence, it is important to remove heavy metals such as Lead (Pb), Zinc (Zn), Copper (Cu) and Cadmium (Cd) which are common heavy metals that can be found in wastewater discharge. Lead, a naturally occurring metal found in the earth. Lead can infiltrate into the water supply through many means such as mining operations and lead water pipes. When lead is absorbed into the body, it causes risk of kidney damage and high blood pressure for adults. At high levels of exposure, lead attacks brain and nervous system causing coma, convulsion and even death. Copper sulfate can infiltrate the water supply through its use in mining industries. As copper sulfate is extremely soluble in water, it can easily seep into water bodies and supplies [2]. Persons suffering from copper poisoning suffer from gastrointestinal issues such as diarrhoea, nausea, abdominal pains and vomiting [3]. Consuming high concentration of copper sulfate can cause multiple organs to fail and death. Cadmium is a naturally occurring metal that can be found in metal ores and as natural deposits in the earth. Cadmium compounds are used in various industries such as pigments in plastics, an anticorrosive layer electroplated onto steel, electrical batteries and components. Cadmium can infiltrate the water supply through zinc pipes and solder containing cadmium that is used to supply water. High levels of cadmium could be found in shellfish and other filter feeding organisms. High amounts of cadmium can damage kidney and cause kidney stones to form and osteoporosis to occur [4]. Zinc can enter the water supply through pipe fittings or zinc pipes used to pipe water. Low pH in tap water can increase the amount of zinc leached into the water. High amounts of zinc can cause nausea, diarrhea, internal bleeding, vomiting and abdominal cramps [5].

Tannery, paper, pharmaceutical and food industries are some examples of industry that produces wastewater discharge with high concentration of heavy metal such as cadmium, lead and other toxic organics. Local authority regulates industries to comply with the standard limit for discharging wastewater into water bodies. However there are still some companies illegally discharging wastewater containing heavy metals and toxic compounds into the water bodies. Continuous incident in Singapore that caused million tonnes of aquatic organisms from rivers in Bishan-Ang Mo Kio, Sungei Buloh, Lim Chu Kang, extending to Changi and Pasir Ris Beach showed an extreme result of coastal water pollution. Many combinations of environmental factors can cause lack of oxygen

and toxicity in water bodies. Plants and aquatic animals will be contaminated and some will be unable to survive except algae. As a consequence, the ability of wastewater treatment system to tolerate and remove toxicity is of considerable importance.

Algae biomass, in general, is an efficient adsorbent of heavy metals. Bio-treatment with microalgae is particularly attractive because of their photosynthetic capabilities, converting solar energy into useful biomasses and incorporating nutrients such as nitrogen and phosphorus causing eutrophication [6]. Also, algae based system for the removal of toxic minerals such as lead, cadmium, mercury, scandium, tin, arsenic and bromine are also being developed [7]-[11].

There are many ways to remove heavy metals from the wastewater, for example, chemical precipitation, ion exchange, membrane, filtration, adsorption using activated carbon and biological techniques [12]. However, these methods are either expensive, or have other disadvantages such as high energy consumption and inefficiencies when the existing heavy metals are in low concentration.

Biosorption can be an alternative method to eliminate heavy metals due to its low cost, high efficiency, minimization of secondary wastes and environmentally friendly. Green algae can be used as natural biosorbent to reduce chemical usage in waste treatment process which indirectly reduces the cost of the treatment plant.

Algae are defined as "thallophytes (plants lacking roots, stems, and leaves) that have chlorophyll as their primary photosynthetic pigment and lack a sterile covering of cells around the reproductive cells". The green algae are distributed in many regions of the world, from being airborne or subaerial to being terrestrial or aquatic, either in freshwater or marine habitats [13]. As an island surrounded by sea, Singapore's algae habitats are found in freshwater and marine water bodies. Therefore, in this project, marine algae (MA) and freshwater algae (MA) biomass are used as heavy metal scavenger in surface waters.

MA, commonly known as seaweed, is large enough to be seen and can grow up to 60 m in length. Types of seaweed include red, brown and green algae. MA do not have vascular system like plants, however they do have pigment chlorophyll for photosynthesis. In addition to the photosynthetic pigmentation, MA has many ultrastructural and biochemical features which include storage compounds, composition of cell walls, presence/absence of flagella, ultrastructure of mitosis, connections between adjacent cells, and fine structure of the chloroplasts [14].

FA is served as a paramount part of pond ecosystem, forming the base food chain. They provide food and energy for fish and other lake organisms and have the ability to absorb nutrients and heavy metals. The most common FA is known as cyanobacteria, contain bright, grass-green pigments. Generally most FA is highly palatable and a good food source for zooplankton [15].

The present study aims to investigate the capability of MA and FA in adsorbing heavy metals such as copper, lead, zinc and cadmium in water medium using spiked deionised water and real water sample from industrial discharge. Characterisation of MA and FA samples was conducted to identify the presence of various functional groups using FTIR analytical instrument. The concentration of all the above mentioned metals was measured using the latest technology of ICP-OES analytical instrument.

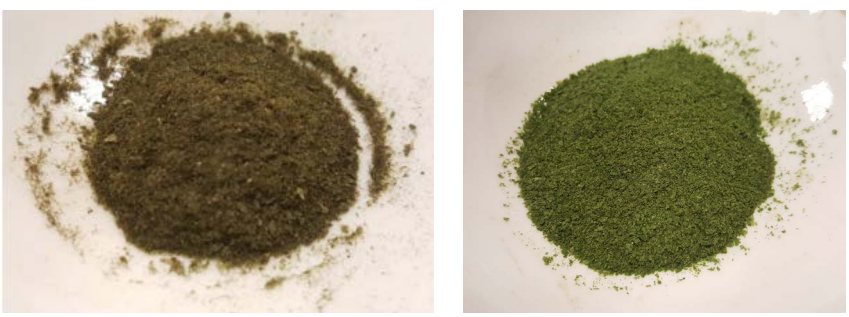
2. Methodology, Equipment and Materials

2.1. Preparation of MA and FA

An estimated mass of about 3 kg of MA was obtained directly from the eastern coast of Pulau Ubin as shown in **Figure 1(b)**. MA was thoroughly washed with tap water for an hour. This was carried out to remove sand and little cretaceous creatures. Foreign particle such as brown algae and dirt that was sticking to the MA was removed manually by hand. It was then soaked in deionized water for about an hour to ensure the cleanness of the samples before being placed in the oven at 104°C for 4- 6 hours to ensure that the samples are completely dried. All the dried algae was then grinded and sieved through a 1 mm^2 net to ensure that all the MA are smaller than 1 mm^2, thus forming a powdered MA. The grinded MA was then stored in labelled glass bottles. An estimated mass of about 8 kg of FA was obtained directly from local fish farm as shown in **Figure 1(a)**. FA was thoroughly washed with tap water to remove some particles such as dirt and leaves which stick and float respectively. Other smaller foreign particles such as duckweed and insects were removed using a tweezer. It was then processed following the above mentioned pre-treatment procedure for MA to form a powdered FA (**Figure 2(a)** and **Figure 2(b)**). The grinded FA was then stored separately in labelled glass bottles. The density of MA and FA were also calculated by weighing the mass of the material in 10 ml of 25 ml measuring cylinder.

(a) (b)

Figure 1. (a) Filtration system of fish farm and FA collection point (shown in red circle); (b) Shoreline of Pulau Ubin beach and MA collection point (shown on red circle).



(a) (b)

Figure 2. (a) Samples of grinded Freshwater Algae (FA); (b) Samples of grinded Marine Algae (MA).

2.2. Characterisation of MA and FA Using FTIR Analytical Instrument

MA and FA biomass characterisation were conducted using FTIR to determine the functional groups of MA and FA samples. No pre-treatment of the MA and FA samples was required due to the fact that the samples were already in powder form of biomass. As such it did not require the use of potassium bromide (KBr) pellets/method. The IR spectra of the samples are recorded by using IRPrestige-21 FTIR spectrophotometer. The spectra were recorded in the transmission band mode in the range of 750 - 4500 nm. Functional groups existing in MA and FA would be shown in the next result section *i.e.* before and after metal adsorption onto MA and FA surfaces.

2.3. Batch Adsorption Experiment Using Synthetic Heavy Metal Solution

0.05 g of FA or MA biomass powder was measured using Sartorius Quintix 224 1S Analytical Balance of accuracy up to four decimal places and was placed directly into the plastic spinning tube. A nominal 30 ml of deionized water was added to each of the plastic spinning tube. The plastic spinning tubes were placed onto the Rotary Mixing Solution YC-80 in the order of 60 min, 45 min, 35 min, 25 min, 15 min, 10 min and 5 min to determine the percentage of metal adsorption at various equilibrium times. Before the appropriate plastic spinning tubes were placed on the rotator, 0.075 ml of 1000 ppm Cu solution was added to each of the plastic spinning tubes to make 2.5 ppm metal concentration. The procedure was repeated for other metals of Pb, Cd, and Zn. All metal standard solutions were purchased from Sigma Aldrich, ICP Analytical Grade of 1000 ppm.

The tubes were rotated at 30 RPM at 45 degrees to ensure full contact and adequate mixing. Once the spinning was complete, the plastic spinning tubes were set to rest for 5 min to allow the MA and FA particles to settle in the bottom of tube. Once the settling time was complete, the solute was extracted and placed into two 15 ml plastic centrifuge tubes. The plastic centrifuge tubes were centrifuged for 3 minute at 6000 RPM to force the algae particle to the side of the plastic centrifuge tube separating the algae particles from the solution. Solution was extracted from the centrifuge tubes using droppers and placed into clean plastic spinning tubes. The remaining concentration of Pb, Cd, Cu, Zn ions in the solution were then measured using the ICP (Thermo Scientific iCAP 6000 Series ICP) at 220 nm, 228 nm, 324 nm, 213 nm respectively. The procedures were repeated twice for each metal ion.

The effect of adsorbent concentration was conducted by adding 0.5 mg, 5 mg, 25 mg, 50 mg, 100 mg, 200 mg and 300 mg added into 30 ml plastic tubes. 0.075 ml of metal standard solution with amount of deionized water was added into the plastic spinning tube, making up to 2.5 ppm of metal concentration. Rotation in the rotary mixer and centrifugation procedures were repeated, except a fixed rotation time of 60 mins. A longer rotation time was set up at 60 mins to fully ensure the metal adsorption process. In a subsequent batch experiment, the effect of pH was conducted by altering the pH of spiked solution with NaOH and HCl, ranging from pH 2 to 11. The abovementioned batch adsorption procedure was repeated, except a fixed rotation set up at 30 mins.

Various metals could exist in one single wastewater discharge. To mimic the real situation, a combined heavy metal was prepared in a subsequent experiment. The above-mentioned procedure was repeated using a combined four metal ions of Cu, Pb, Cd, Zn in a deionised water to understand the capacity and preferential metal adsorption onto MA and FA surfaces. Each metal was made 2.5 ppm, resulting a total metal concentration of 10 ppm from the four selected metals.

2.4. Batch Adsorption Experiment Using Real Industrial Water

A real industrial water was collected from industrial area of metal manufacturing factory. Fresh wastewater discharge after membrane wastewater treatment was collected using 1 L plastic bottles and stored and labelled in laboratory. Using the batch adsorption procedure in Subsection 2.3, all the metal concentration was measured using ICP-OES at once after two days of the settlement of larger particles. This is to reduce a filtration step process before measurement using sensitive equipment of ICP-OES, where the dissolved metals only could be measured.

3. Results and Discussion

3.1. MA and FA Biomass Characterisation Using FTIR

MA had a density 488.04 μg/mL, which was measured approximately 2.5 times denser than FA. Figure 3 showed a scanned wave numbers from 4000 cm^{-1} to 1000 cm^{-1} from

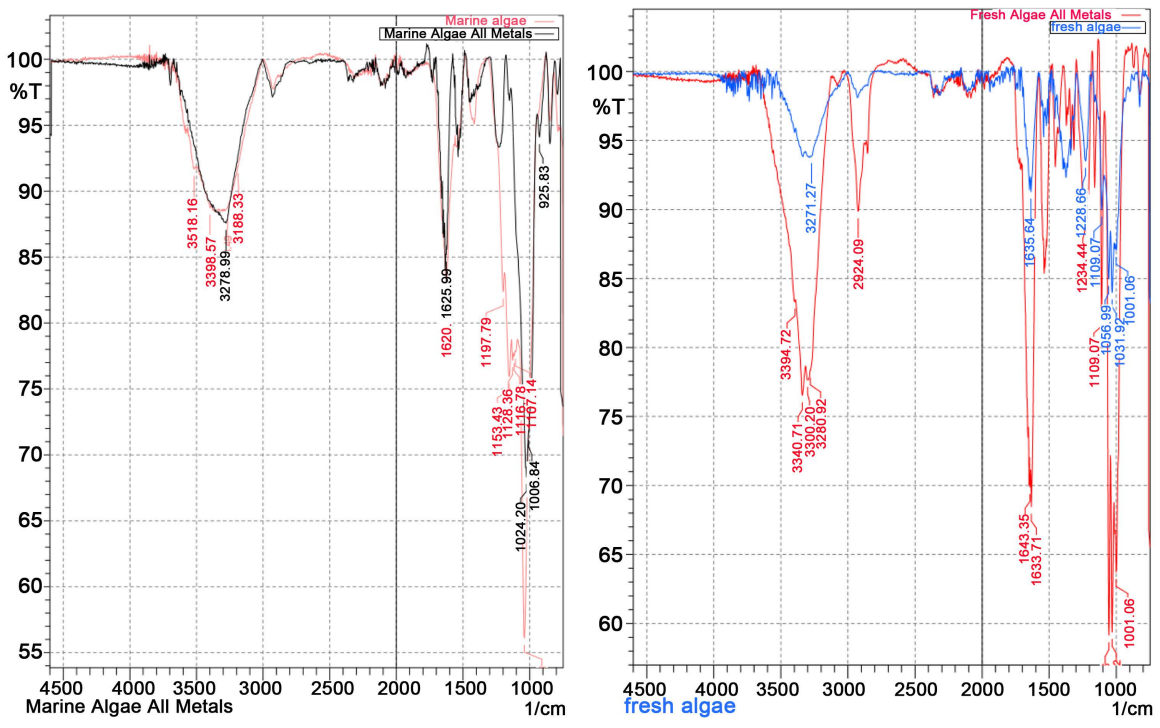


Figure 3. (a), (b): FTIR spectra of MA (a-left) and FA (b-left) samples. Intensity changes are shown at different wave length before and after heavy metal adsorption onto MA and FA surfaces.

both FA and MA, before and after exposure to a 30 mL solution containing Cu, Pb, Zn and Cd at concentrations of 2.5 ppm. The FTIR analysis demonstrated which functional groups will be responsible for the adsorption of the metals present in the solution. Figure 3(a) shows FTIR spectra of MA sample with the bands associated with the hydroxyl O-H group, lignin and acetyl group. The bands at 3300 cm^{-1} and 2900 cm^{-1} are assigned to O-H stretching and C-H stretching respectively, and it exist in both MA and FA samples. MA had higher intensity at 3300 cm^{-1} (O-H stretching) than FA, however after all the four heavy metals adsorbed, FA showed the opposite result of higher O-H stretching. No significant intensity change after heavy metal adsorbed onto MA. The result showed that carboxylic functional group may be responsible for the heavy metal adsorption onto FA surface. A similar trend was also observed after the metals were bonded in the band 2900 cm^{-1} (C-H stretching). The band at 1600 cm^{-1} is assigned to C-C stretching in FA after the metals being adsorbed with no significant intensity change in MA. At the wavelength of 1000 cm^{-1} (C-O and C-N stretching), MA showed a large intensity change indicating that the heavy metals were adsorbed on the MA surface. The FA also showed similar significant results as MA. The result showed that the carboxylic and amine functional groups may be responsible for the heavy metal adsorption onto both MA and FA surfaces.

3.2. Effect of Adsorption Time

Figure 4 shows that FA was able to adsorb more Zn compared to other metals of Cu, Pb and Cd in separate solutions. The adsorption process showed a very fast adsorption process taking place in minutes and reaching a plateau after 20 minutes. Therefore in the subsequent analysis, effect of pH and concentration of adsorbent, a minimum fixed time of 30 mins would be used. FA adsorbed about 45 mg of Zn in each gram of FA. The reaction was instantaneous, removing almost 45 mg of Zn per gram of algae present

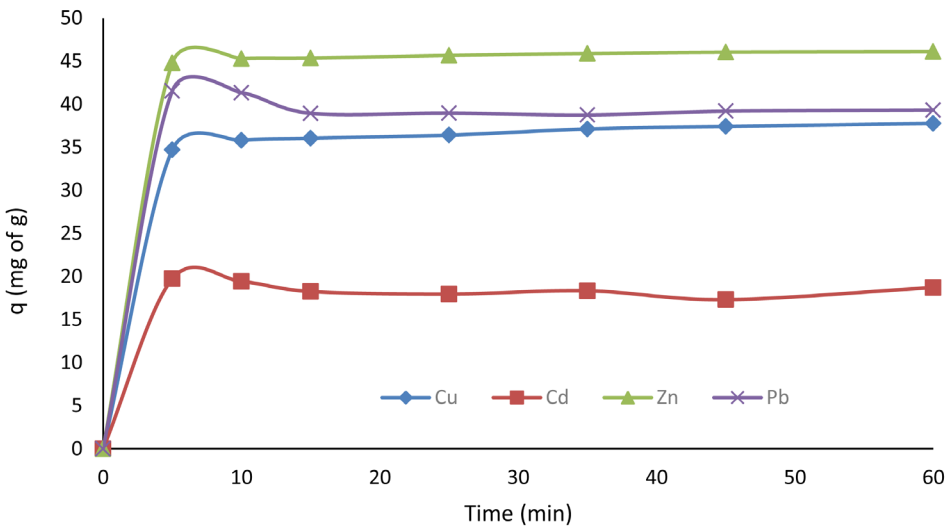


Figure 4. Adsorption kinetics of Cu, Pb, Cd, Zn onto by 0.05 g of FA. Initial concentration of metal is 2.5 ppm

within 5 min, it remains constant throughout the remaining adsorption time. FA was also able to adsorb Pb, Cu and Cd at lower concentration of 38 mg/g, 35 mg/g and 17 mg/g of FA respectively, measured after 30 mins. Alternatively, the percentage of adsorption onto 50 mg of FA could be calculated as 91.76%, 77.49%, 74.24% and 36.68% for Zn, Pb, Cu and Cd respectively.

Figure 5 shows MA was able to adsorb more Zn and Cu compared to other metals within 60 min. MA was able to adsorb about 42 mg/g of Zn within 60 min. It could also be observed that the percentage of heavy metal adsorbed remained constant throughout the entire 60 min. The reaction was also instantaneous adsorbing almost 43 mg/g of the Zn present within 5 min, the amount of heavy metal adsorbed started to decrease as the exposure time increased. The amount adsorbed decreased by about 1 mg/g. Since the adsorption did not have a significant change, it was considered that the reaction plateaued after 5 min. A similar trend was observed for Cu. As for Pb, MA was able to adsorb about 40 mg/g of the Pb within 60 min. However unlike the other metals, the amount adsorbed continued to increase at a steady rate as the exposure time increased, from 30 mg/g at 5 min to a stable rate of 40 mg/g at 60 min. Therefore the optimum time for Pb was considered after 60 mins. Cd is the least adsorbed metal by MA within 60 min, similar trend as observed for FA when adsorbing same Cd as the contaminant. MA was able to adsorb about 16 mg/g of Cd present within 60 min. The reaction was instantaneous, adsorbing 15 mg/g of the Cd present within 5 min. For simplicity, a fixed adsorption time of 60 minutes would be used in the subsequent experiments. Alternatively, the percentage of adsorption onto 50 mg of MA could be calculated as 84.52%, 84.38%, 78.84% and 33.24% for Zn, Cu, Pb and Cd respectively. The change of order of Cu and Pb metal ions adsorbed onto MA and FA showed that different functional groups, which exist on the surface of FA and MA, might have a preferential process of adsorption. Following the discussion in subsection 3.1, the result may be linked up that FA had a couple of functional groups, which may increase the heavy

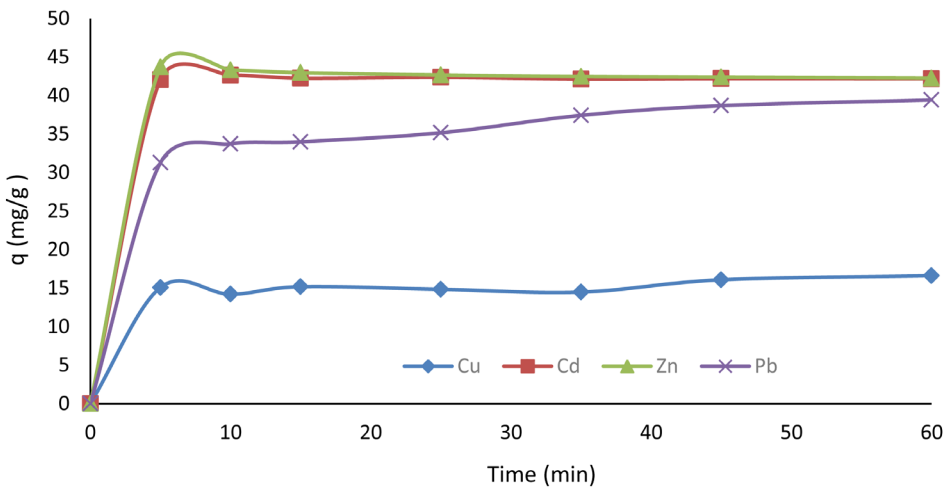


Figure 5. Adsorption kinetics of Cu, Pb, Cd, Zn onto by 0.05 g of MA. Initial concentration of metal is 2.5 ppm

metal adsorption rate. Meanwhile, MA had a singular large intensity change after the heavy metals being adsorbed at the wavelength of 1000 cm^{-1} due to carboxylic and amine functional groups.

Figure 6 shows the result of batch adsorption experiment using the combined four metals of Cu, Pb, Zn and Cd with the total metal concentration of 10 ppm. An instantaneous adsorption process was also observed in Figure 7. Pb is the most adsorbed metal compared to the other metals, followed by the Cu, Cd and Zn, which was the least. FA was able to adsorb about 23 mg/g, 10 mg/g, 9 mg/g and 7 mg/g of Pb, Cu, Cd and Zn respectively. FA's adsorption capability on each metal from the combined four metals solution showed a lower rate of about 50% - 70% than the previous FA's

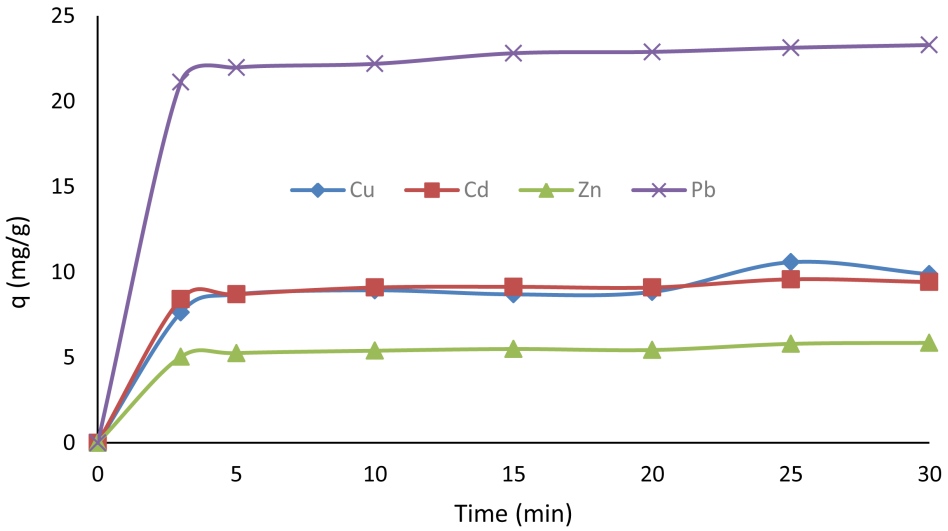


Figure 6. Adsorption kinetics of combined Cu, Pb, Cd, Zn onto by 0.05 g of FA. Total metal concentration is 10 ppm with equal concentration of each metal of 2.5 ppm.

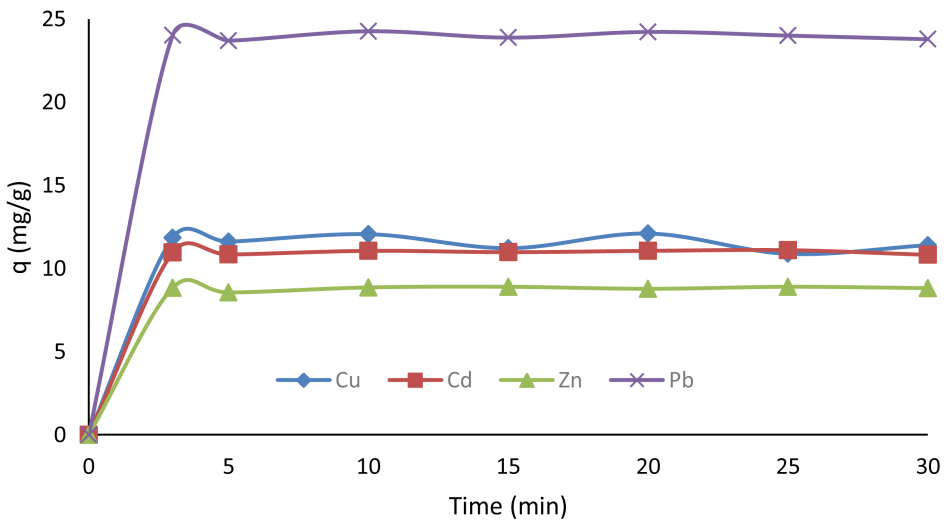


Figure 7. Adsorption kinetics of combined Cu, Pb, Cd, Zn onto by 0.05 g of MA. Total metal concentration is 10 ppm with equal concentration of each metal of 2.5 ppm.

adsorption capacity on each metal from individual metal solution. The result meant that the adsorption was technically limited to the availability of FA surface area with the active functional groups.

Figure 7 shows that similar fast adsorption process was also shown for MA with the order of adsorbed metals of Pb, Cu, Cd and Zn of 25 mg/g, 11 mg/g, 10 mg/g and 8 mg/g respectively. Since all the metal adsorption fluctuates at a difference of 1 mg/g, it could be considered constant. This means that MA achieved its maximum adsorption within 3 min of exposure, therefore the reaction was considered instantaneous. MA's adsorption capacity using combined four metals also showed a lower rate of about 50% - 70% than MA's adsorption capacity using individual metal.

The result showed a confirmation that adsorption process was limited to the availability of FA surface area with the active functional groups. Figure 6 and Figure 7 showed that both MA and FA were able to adsorb Pb more readily compared to the other metals and Zn is the least adsorbed metal.

3.3. Effect of MA and FA Adsorbent Concentration

An increasing amount of MA or FA may be able to fully adsorb heavy metals or reach an equilibrium state when reaching a plateau at a fixed concentration of metal. Percentage of each metal at fixed concentration of 2.5 ppm was adsorbed by FA and MA as presented in Figure 8 and Figure 9 respectively.

Figure 8 and Figure 9 showed that the increasing mass of FA and MA were able to adsorb considerable percentage of metal in water. Figure 8 showed that FA was able to adsorb Pb, Cd, Zn and Cu at nominal percentage of 94%, 75%, 86% and 53% respectively at 300 mg dosage. FA showed a lesser increment of percentage of adsorption on Cu rather than on Cd and Zn after 50 mg of FA. Figure 9 showed that MA was able to adsorb Pb, Cu, Cd and Zn at nominal percentage of 92%, 64%, 61% and 53% respectively at 300 mg dosage. MA adsorbed all the four metals at a constant rate of adsorption

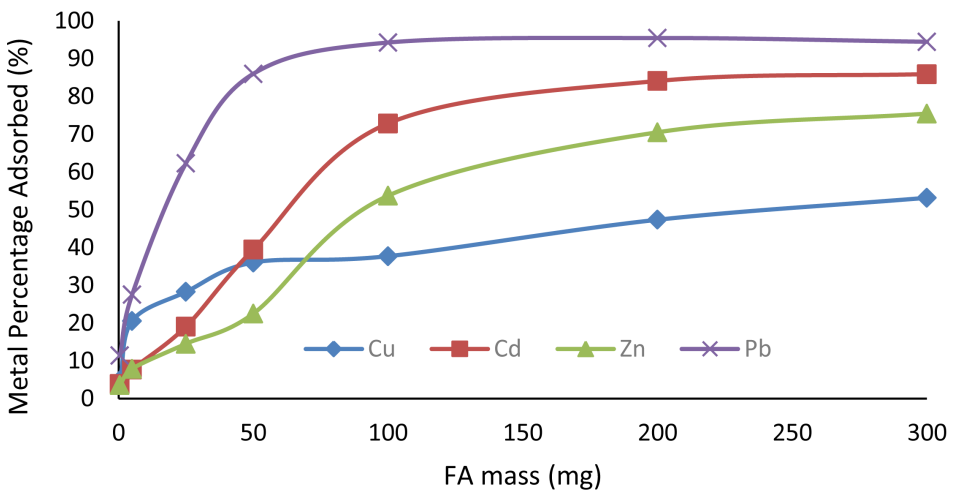


Figure 8. Percentage of metal of Cu, Pb, Cd and Zn onto FA. Initial concentration of each metal is 2.5 ppm.

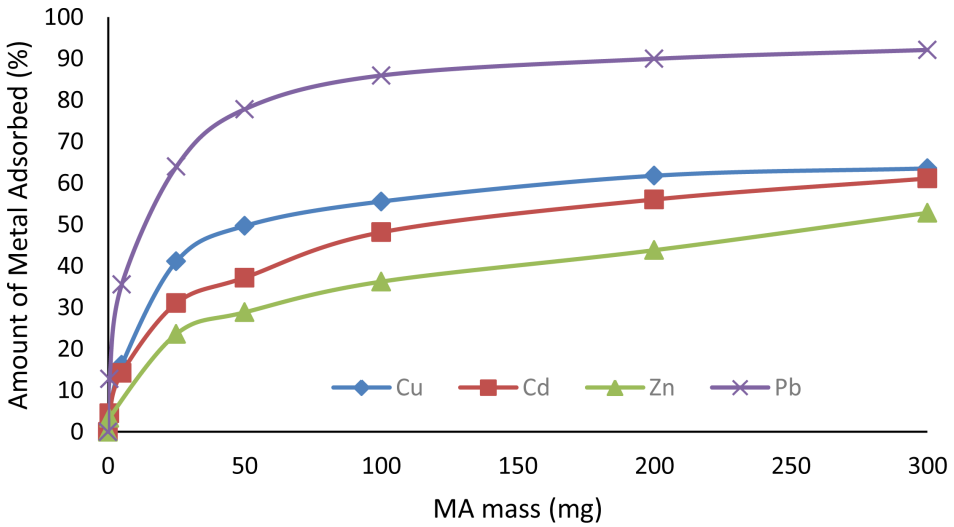


Figure 9. Percentage of metal of Cu, Pb, Cd and Zn onto MA. Initial concentration of each metal is 2.5 ppm.

until reaching plateau after 200 mg of MA dosage. Both FA and MA showed the dried algae have a strong percentage of adsorption on Pb at 94% and 92%.

3.4. Effect of pH on Heavy Metal Adsorption onto MA and FA

Figure 10 shows that the optimum pH for adsorption of heavy metals for FA is between pH of 5-pH of 10. It shows that the highest adsorption rates occur at pH 5 with the exception of Cd, which was most readily adsorbed at pH 11. It can be concluded that Pb was the highest adsorbed metal while Cu was the lowest adsorbed metal.

Figure 11 shows that the optimum pH for adsorption of heavy metals for MA also lies between pH 5 - pH 10. This is due to the plateau observed within the pH 5 - pH 10 range. Highest adsorption rates also occurred at pH 5. It is also concluded that Pb was the highest adsorbed metal while Zn was the lowest adsorbed metal.

3.5. Batch Adsorption Experiment Results Using Real Industrial Water

Figure 12 shows that the industrial water contained significant concentration of Cu and Zn dissolved ions, up to 10.425 ppm and 30.835 ppm respectively, and very minimum concentration of Cd and Pb metal ions. At a fixed equilibration time of 30 mins, MA showed a higher adsorption rate capacity for all metals than FA did. The result proved that FA and MA had a higher capability in adsorbing a total metal of about 40 ppm level from an industrial water, or 4 times than synthetic water concentration, at the same adsorbent dosage of 50 mg. Moreover, Zn was adsorbed at 6.87 mg/g and 7 mg/g of FA from an industrial water and synthetic water respectively. Similarly, Zn was also adsorb at 7.17 mg/g and 8 mg/g of MA from industrial water and synthetic water respectively. MA has higher adsorption of all metals when compared to FA. MA adsorbed 22%, 67%, 98%, 39% of Cd, Cu, Pb and Zn respectively, while FA adsorbed 18%, 29%, 94%, 37% of Cd, Cu, Pb and Zn respectively.

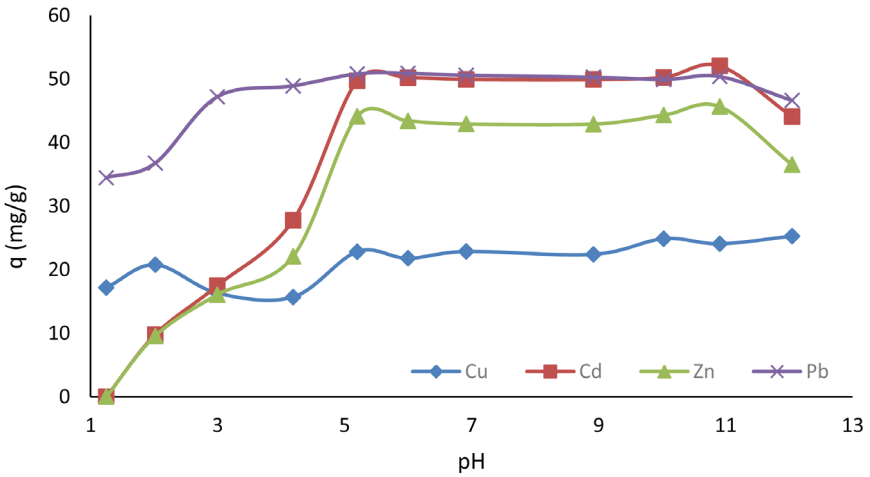


Figure 10. Adsorption rate of metal of Cu, Pb, Cd and Zn (mg/g) onto FA at a fixed time of 30 mins. Initial concentration of each metal is 2.5 ppm.

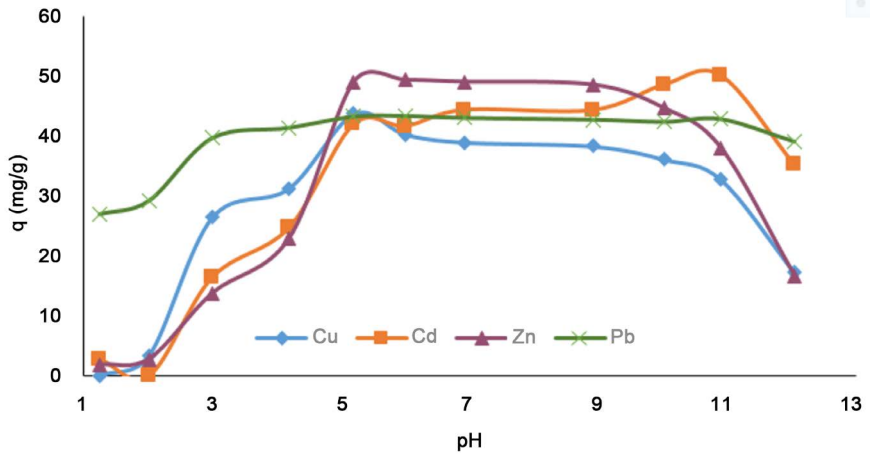


Figure 11. Adsorption rate of metal of Cu, Pb, Cd and Zn (mg/g) onto MA at a fixed time of 30 mins. Initial concentration of each metal is 2.5 ppm.

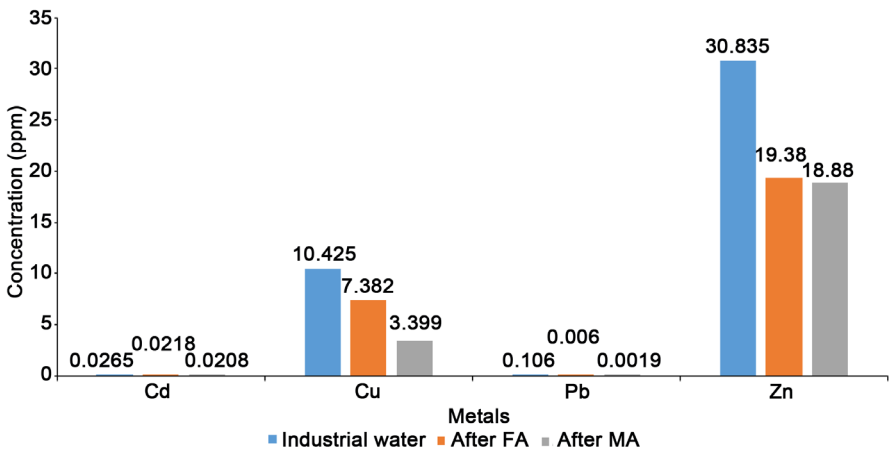


Figure 12. Comparison of metal concentration, before treatment (left bars—blue colour) and after treatment using FA (middle bars—orange colour) and MA (right bars—grey colour).

4. Conclusion

The presence of various functional groups, hydroxyl, carboxyl and amine groups, in all MA and FA samples had enabled the dried algae to adsorb heavy metals of Cu, Pb, Cd and Zn from industrial and synthetic water. Due to their biosorptive properties and fast adsorption capability, algae biomass could be a potential method for cleaning up surface water or post-treatment of wastewater. The MA and FA biomass have capability to adsorb heavy metals at intermediate pH, ranging from pH 5 to 11. Overall, use of algae biomass for heavy metal scavenger can offset the cost of eutrophication.

Acknowledgements

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