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Original Research Paper

Adsorption of iron by using hybrid *Akar Putra* and commercialized chicken eggshells as bio-sorbents from aqueous solution

***H.M. Nasir*[1], *A. Azmi*[1], *A.Z. Aris*[1,2,*], *S.M. Praveena*[3]**

[1]*Department of Environmental Sciences, Faculty of Environmental Studies, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor Malaysia*

[2]*Laboratory of Halal Science Research, Halal Products Research Institute, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia*

[3]*Department of Environmental and Occupational Health, Faculty of Medicine and Health Sciences, Universiti Putra Malaysia 43400 UPM Serdang, Selangor Darul Ehsan, Malaysia*

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ABSTRACT: Heavy metal contamination in the environment could cause harmful effects both to human health and aquatic life. Numerous remediation methods had been developed to encounter with the contamination problem prior to degrade, decrease and to purify the contaminated water at minimal concentration as low as possible. Therefore, in current study, commercialized chicken eggshells and hybrid *Akar Putra* chicken eggshells were conducted in batch experiment to testify the capabilities of bio-sorbent materials in iron (II) ion removal from aqueous solution at optimized level of dosage and equilibrium contact time. The optimum condition for iron (II) removal for commercialized chicken eggshells and hybrid *Akar Putra* chicken eggshells bio-sorbents reached at 0.30 g with optimum contact time of 50 minutes and 91.83% and 91.07% of removal percentage with 0.60 g at 40 minutes. The final concentration from both bio-sorbents is achieved below than drinking water guideline (0.30 mg/L), 0.1635 mg/L and 0.1785 mg/L, respectively. The isotherm adsorption results showed it fitted better in Langmuir Isotherm Model than in Freundlich Isotherm Model, however with weak bonding, which could not held onto the heavy metal ions in long time period. In brief, commercialized chicken eggshells and hybrid *Akar Putra* chicken eggshells have considerable potential in removing heavy metal in aqueous solution. The selection of the bio-sorbent materials is more favorable as it reduces dependency towards chemical usage in water treatment which could have complied with drinking water guideline that can be obtained easily, abundance in amount, cheap and biodegradable.

KEYWORDS: *Adsorption; Bio-sorbent; Contact time; Dosage; Isotherm; Guideline*

INTRODUCTION

Heavy metal pollution is referred as any irresponsible release of metallic element in water bodies, air and sedimentation whereby it can cause toxicity or poisonous effect even at low concentration (Lim *et al.*, 2012). Although some of the heavy metals present in the environment naturally, anthropogenic activities are the main causes that lead to sudden rise in the concentration of heavy metal pollutants that could finally affect the present quality of the environment beyond tolerance limit (Al-Badaii and Shuhaimi, 2014; Prasanna, *et al.*, 2012; Sujaul *et al.*, 2013; Looi *et al.*, 2016). The vast majority of researcher found the

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*Corresponding Author Email: *zaharin@upm.edu.my*
Tel.: +603 8946 7461; Fax. : +603 8943 8019
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contamination of heavy metal in the river water bodies which can be easily be identified like cadmium, manganese, nickel, cobalt, chromium), copper, lead, iron, mercury, uranium, selenium, zinc, arsenic, silver (Alhaya *et. al.,* 2003; Lim *et al.,* 2012; Prasanna *et. al.,* 2012; Sujaul *et.al.,* 2013; Al-Badaii *et al.,* 2013) that discharge from the industrial sector. These sectors allow the effluent even without proper treatment and could contribute to various heavy metals pollution to the river water bodies that need the high cost of treatment.

Heavy metals like iron which can be found naturally that present widely in daily foods such as variety of meats and vegetables, which are more easily absorbed in the body through meat ingestion (Lenntech, 2015). The requirement of iron in the body are in different intake based on the genders and ages whereby infants and children d" 8 years old required in average about d" 10 mg/day. Meanwhile males and females e" 8 years old need in average about 8 mg/day. In fact, the iron systematic balancing of iron in the body is depending on the signaling mechanisms in the body known as hepcidin-FPN which responsible for iron supply, utilization, recycling and storage (Guo *et. al.,* 2015). However, human body requires an appropriate iron level intake as agents to transport oxygen throughout the whole body in hemoglobin. Other than that, it is usually being applied and used widely in various purposes for industries, human and animals supplement also as plants additives supplements (Ankerpoort, 2015). Therefore, iron homeostasis in blood circulation has to be in a controlled amount, as it is capable to animate the generation of radicals, which intrude and disrupt macromolecules in, cellular by promoting cell death and tissue impairment (Papanikolaou and Pantopoulos, 2005). However, excessive iron inhalation and ingestion would cause chronic diseases such as pneumoconiosis also lung cancer. Moreover, the attachment of excessive iron in environment in the manner of iron(III)-o-arsenite and iron(III)-o-pentahydrate that persistent, hazardous and could be affects the eco-balance have some adverse effects and damages beyond imagination towards health problem without seeking solution for removal (Tinaroth, 2012). According to Torti and Torti (2011), the excessive iron bioaccumulation either in the liver, tissue and other human body organ can allow development to malignant transformation and hepatocellular cancerous cells.

There are several approaches that had been extensively used in removing heavy metal from the water and wastewater such as chemical coagulation, electro-chemical precipitation, electro-dialysis, ion exchange, membrane filtration, oxidation, phytoremediation, reverse osmosis, and ultrafiltration. Most of these processes can be unfavorable choices with their own disadvantages due to high cost, produces large and toxic sludge which is undesirable end results (Basha *et al.,* 2007), meanwhile ion exchange method is not practicable as the cost of the synthetic ion-exchange resins could be costly (Ipeaiyeda and Tesi, 2014). Therefore, bio-sorption is preferable approach as the bio-sorbent that used in the adsorption process are inexpensive (Lim and Aris, 2014a;b), easy availability with collection from the food-processing production (Bailey *et al.,* 1999; Jain *et al.,* 2013; Lim and Aris, 2014a) and high efficiency removal of heavy metals from water (Zahra, 2012).

Majority of bio-sorption experiments that have been conducted throughout years either in removal of oil, lignosulfonate compounds, basic dye, methyl orange, toxic metals: copper, zinc, or even recoveries of precious metal (Won *et al.,* 2014). Several studies have been conducted based on the interest of the bio-sorbents materials which contains calcium carbonate such as: dead calcareous skeleton (Lim and Aris, 2014b); seashells (Chowdhury and Saha, 2012); bone charcoal (Wilson *et al.,* 2003); clam, oyster, lobster (Tudor *et al.,* 2005); shrimp shell; fish scales, coral which some of these bio-sorbents used can removed heavy metal up to 200 mg/L. Besides, there are also several other household waste and industrial waste that been widely used as bio-sorbent materials such as rubber, *Hevea brasiliensis* leaf powder (Kamal *et al.,* 2009); banana peel, pumpkin peel, eggshell (Kanyal *et al.,* 2015); tea waste (Thakur and Parmar, 2013) and etc. These materials shown good removal of heavy metals, however, eggshell is chosen in this experiment, as this material is eco-friendly, cost effective, widely available in huge amount from the food waste and industrial chicken livestock production waste (Lim and Aris, 2014a).

Bio-sorbent that being used must possess the high capability in adsorption and desorption in order to be able to treat heavy metal more efficiently in a shorter time. Commercialized chicken eggshells (CCE) and hybrid *Akar Putra* chicken eggshells (HAPCE) were chosen as the bio-sorbents materials in heavy metal removal from aqueous solution, due to its abundance sources (farm, household and industrial waste), water solubility, high surface of homogeneity which allow metal ions and large organic nutrient cross-linked with protein molecules (Cordeiro, 2015), low cost and biocompatibility properties. The aim of this study were to investigate the potential of CCE and HAPCE as bio-sorption in Fe removal at optimize level of dosage and contact time in equilibrium condition. The main goal in this study is to achieve the Fe removal by using CCE and HAPCE comply with drinking water guideline and applying the data in isotherm models. The present findings of Fe are above the drinking water and river water level requirement which is 0.3 mg/L and 1.0 mg/L which it can caused toxicity to human bodies and aquatic organism even though it present in low dosages. Although it is well known that presence of little iron in the water bodies is required as a bioavailability micronutrient to certain organisms and plants, however, when excess of the iron occurred, it poses threat to the organism's cells and tissue. Thus, monitoring of amount of heavy metal by following the mandatory standard of drinking water and river water quality standards to remove Fe is important to provide a secure and healthy lifestyle to the human living and aquatic life which depending on the water which is part of living things' needs. This study has been performed in Environmental Forensics Laboratory of Halal Products Research Institute, Universiti Putra Malaysia in 2015.

MATERIALS AND METHODS

Materials

Hybrid *Akar Putra* chicken eggshells (HAPCE) were obtained from chicken barn at Universiti Putra Malaysia, Serdang, Malaysia and commercialized chicken eggshells (CCE) were collected by selected restaurant as food waste in Serdang. Ferrous (II) sulphate heptahydrate (Sigma Aldrich, Switzerland), deionized water from Milli-Q system (Millipore Corp, USA) and concentrated nitric acid, HNO_3, 65% (Merck, Germany) were used as chemicals. The initial pH of synthetic solution was tested by using pH meter (Orion 2 Star, USA) and centrifuge machine (Rotofix 32, Zenttrigugen, Germany) for separation liquid and solid phase function. The concentration of iron was analyzed by using flame atomic adsorption spectroscopy (FAAS; AA-01-0440, Perkin Elmer, Canada). The laboratory settings for this research has been performed in Environmental Forensics Laboratory from June – September 2015.

Methods

Adsorbate and adsorbents preparation

Stock solution of 1000 mg/L of ferrous (II) ion was prepared from Ferrous (II) sulphate heptahydrate, $FeSO_4.7H_2O$ before diluted into several concentrations. The membrane HAPCE was manually peeled off and soaked in water bath for 30 minutes prior rinsed with distilled water. HAPCE and CCE were rinsed again with hot and distilled water repeatedly to remove impurities such as oil and sticky food waste on the surface of the commercialized chicken eggshell. Then, dried at 70°C for 4 hours (Nezhad *et al.,* 2016) followed by crushing step and sieved into the fraction size, ≤900 µm (Flores-cano *et al.,* 2013) then kept in acid wash zip lock bag for directly used in further analysis (Chowdhury *et al.,* 2013).

Batch experiments

The batch adsorption experiment was set up at room temperature by using two different types of eggshell bio-sorbent at varying weight of eggshell bio-sorbent dosage 0.15–0.90 g were added to 50 mL of iron metal synthetic solution to produce several targeted concentrations, ranging from 0.5-2.5 mg/L. The experiments were conducted at time interval of 180 minutes at initial pH in constant turbulence speed of 150 rpm orbital shaker. After being shaken, the solution separated was withdrawn using syringe and filtered with 0.45 µm nylon-membrane filters and the equilibrium of iron concentrations were analyzed by AAS. The calibration curve was prepared by using the iron standard in several concentrations ranging from 0.5 to 2.0 mg/L prior the samples analysis to create the ideal calibration curve for detecting iron element. The validation method was analyzed by using the optimized value of bio-sorbent dosage and equilibrium contact time. The obtained results with q_e value, quantity of metal adsorbed at equilibrium (mg/g) and the adsorption percentage, % was calculated by Eq. 1.

$$qe = \frac{(Ci - Ce)\,V}{m} \tag{1}$$

where Ci and Ce are the initial at equilibrium metal ion concentration in mg/L;

qe represents the amount of heavy metal ion adsorbed onto adsorbent per weight (mg/g);

V is the solution volume that being treated and m as mass of adsorbent. The experimental data obtained were analyzed using Langmuir and Freundlich isotherm model.

RESULTS AND DISCUSSION

Analysis of heavy metals by Flame Atomic Adsorption Spectroscopy (FAAS)

FAAS measures the concentration targeted elements in environmental samples by the adsorbed radiation. FAAS detector measures the light transmitted by standard solution and compared with the originally samples passed through with specific wavelength. FAAS operation conditions are shown in Table 1. The standard solutions were prepared for the analysis of Ferrous (II) ion concentration by FAAS. Several concentrations with 0.5, 1.0, 1.5, 2.0 mg/L were prepared freshly with Milli-Q water. Concentration vs absorbance is plotted in Fig. 1. Satisfactory values were obtained with r = 0.9999 and the recovery for the analyses were in the range of 100% ± 5% (95-99%).

Potential of CCE and HAPCE as bio-sorbent in Fe removal at optimum level of dosage and contact time in equilibrium condition

The isotherm experiments had been carried out with several dosage of bio-sorbent, 0.15-1.5 g with constant temperature, initial pH and initial concentration of synthetic solution, 2.0 mg/L. The selection of contact time range was chosen according to Jai *et al.* (2007) and Ipeaiyeda and Tesi, (2014). Findings revealed that most of the contact time equilibrium reached within early 20 until 75 minutes. The contact time was prolonged to have a better view of the equilibrium curve.

The results shown both of bio-sorbents dosage of CCE and HAPCE illustrates immediate reduction in Fe concentration with initial 2.00 mg/L to lowest and high concentration, whereby CCE bio-sorbent 0.19 mg/L, 0.9 g bio-sorbent dosage and 0.86 mg/L, 0.15 g and HAPCE bio-sorbent 0.14 mg/L, 1.2 g and 0.98 mg/L, 0.15 g of bio-sorbent (Fig. 2). The concentration of Fe after treatment with different HAPCE and CCE dosage is illustrated in Fig. 2. It shown that HAPCE have weak adsorption interaction ($p<0.05$) between heavy metal ions in the aqueous solution. Moreover, from the pattern of the trend line, it shown that, metal ion that attach on the surface of the eggshell cannot be hold in a long time of period more than 10-20 minutes. Therefore, the CCE bio-sorbent with removal dosage efficiency at equilibrium time listed as followed 0.15 g < 1.2 g< 1.5 g< 0.45 g < 0.60 g < 0.90 g < 0.30 g. For HAPCE, the removal dosage efficiency at equilibrium time is 0.15 g < 0.30 g < 0.45 g < 0.90 g < 0.60 g < 1.2 g for HAPCE bio-sorbent. Generally, both of bio-sorbent types have a fluctuated tread line in iron removal, however the constant and more stable tread line with small dosage at faster contact time of CCE and HAPCE found at 0.30 g at 50 minutes and 0.60 g at 40 minutes, respectively. The fluctuated reading, which may be caused by unstable adsorption of metal ion by the eggshell or can be due to reaching early short time period of equilibrium where de-sorption taken placed rapidly after the adsorption phase.

Table 1: FAAS Operation conditions

Parameters	Iron
Wavelength	248.3 nm
Lamp current	75%
Signal	Continuous
Fuel flowrate	0.9 L/min
Run time	4 sec
Recovery	95 - 99%

Comparison of Fe removal for CCE and HAPCE bio-sorbent

Comparison both of the bio-sorbent types were used of the optimization values to comes out with the highest removal efficiency. CCE and HAPCE found that have different equilibrium rate in term of contact time and adsorbent dosage. Based on the optimization values of the contact time, CCE that have 10 minutes more rather than HAPCE shown that the small dosage, 0.30 g needed to remove up to 91.83% compared with HAPCE that required 0.60 g of bio-sorbent dosage which is much higher than the amount that being required by CCE to achieved up to 91.07% of Fe removal. The results found an almost similar removal trend ($p<0.05$) for both bio-sorbent types.

As the removal efficiency depending on shortest contact time which can remove iron concentration in the aqueous solution up to drinking water quality guideline requirement which is below than 0.3 mg/L (WHO, 2011) . Current findings were in agreement with

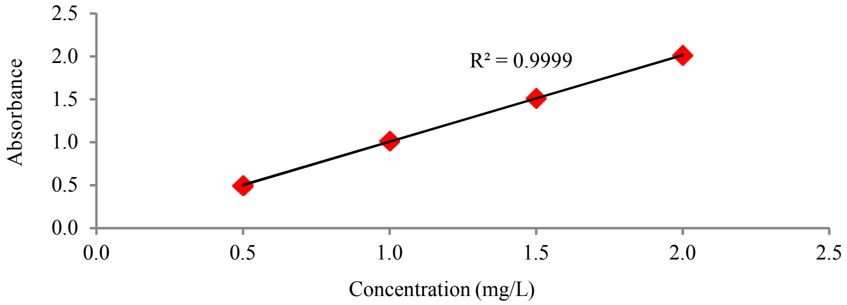

Fig. 1: Calibration curve for Iron by FAAS

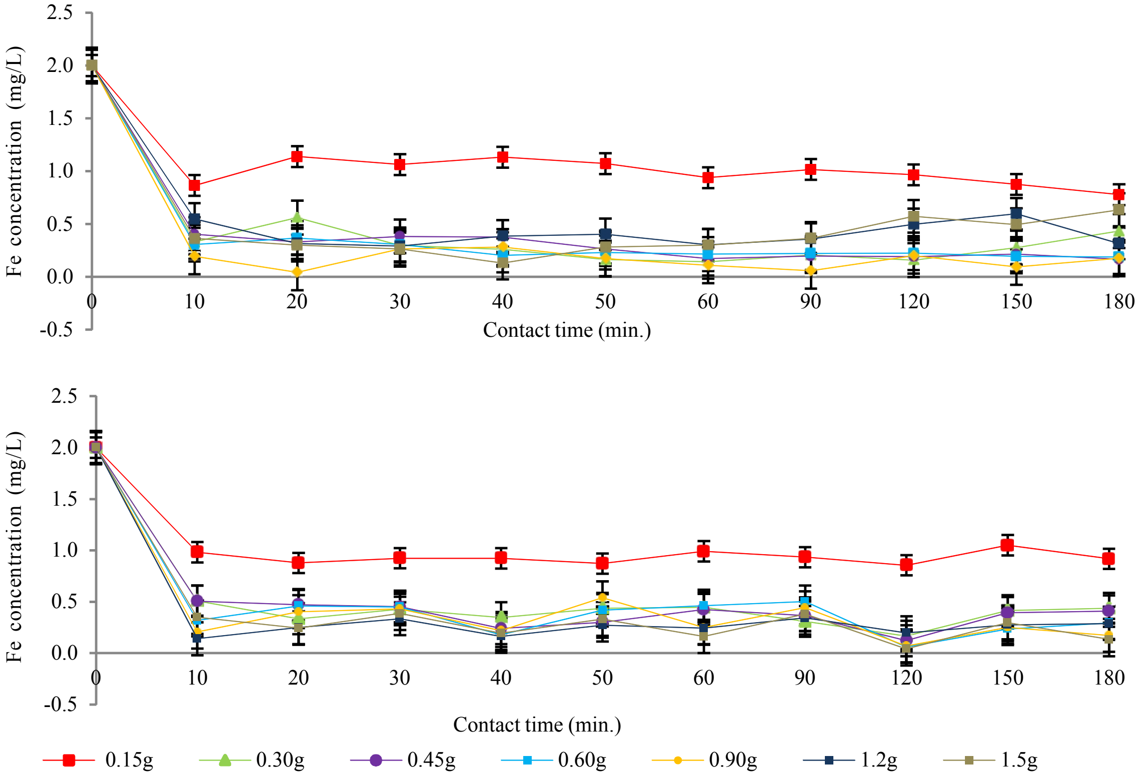

Fig. 2: Iron (II) removal for several dosages (a) CCE (b) HAPCE

study conducted by Vijayaraghavan *et al.* (2006) where higher biosorbent dosage will produced the higher adsorption capacity. In contrast of CCE bio-sorbent which followed partially of the theory of higher amount of dosage present more available site to be attached with and thus more adsorption can be achieved (Rao *et.al*., 2010). This is due to at first 10 minutes of contact time; the highest efficiency is achieved by CCE with 0.90 g amount but not with the highest amount 1.5 g. However, the final results for both of bio-sorbent types shown the final concentration Fe below than guideline whereby remain concentration of CCE is 0.1635 mg/L and also below than 0.3 mg/L for HAPCE with 2.0 mg/L as initial concentration of synthetic solution considered as the concentration before treatment.

The validation method was used optimized value of bio-sorbents dosage and equilibrium contact time from CCE and HAPCE were used to re-affirm the efficiency portraits. Fig. 3 illustrated percentage removal of Fe for each 0.30 g and 0.60 g of CCE and HAPCE whereby CCE view more stable with no significant difference ($p>0.05$) in high removal efficiency condition compare to HAPCE bio-sorbent. Based on the experiments, it has concluded that the CCE bio-sorbent has better removal efficiency than HAPCE in Fe aqueous solution whereas both of the final concentration after treatment below than drinking water guideline of WHO, (2011) as shown in Fig.3.

Adsorption isotherms

Adsorption isotherm basically described the interaction of adsorbate ion and adsorbent used in the aqueous solution, the remaining metals ions in the aqueous solution during surface bio-sorption and to present the equilibrium that can achieved (Feng *et. al.*, 2010). These adsorption isotherms were used to calculate the maximum adsorption capacity of bio-sorbent types based on the optimized values of dosage and contact time. Langmuir and Freundlich isotherm model are the isotherms, which are broadly applied for bio-sorption experiment. Therefore, the fundamental process in adsorption isotherm is to determine the minimal capacity of adsorption towards the metal ions (Larous *et al.,* 2005). Langmuir isotherm model shown the nature adsorption of CCE and HAPCE as favorable categories whereby

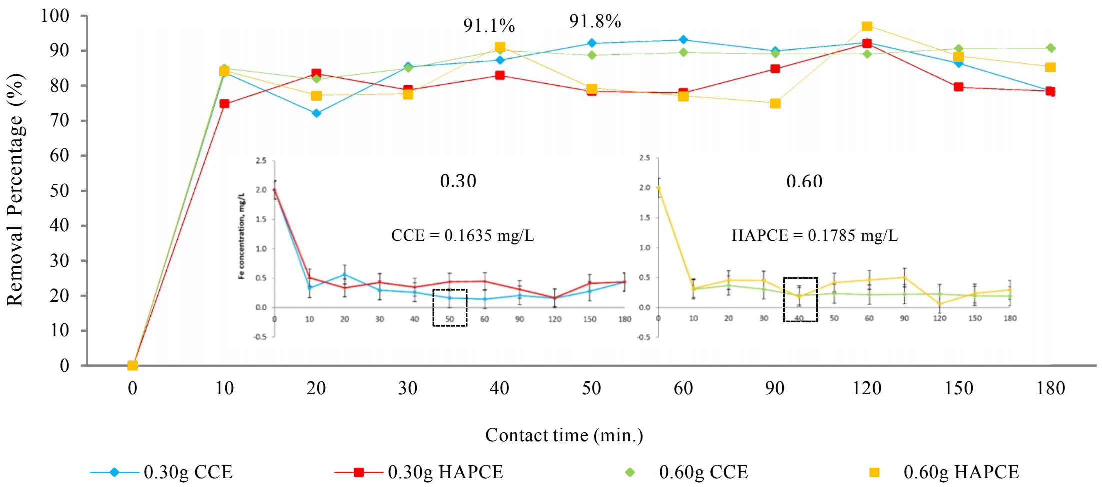

Fig. 3: Iron (II) percentage removal for optimized dosage and contact time with final concentration of aqueous solution

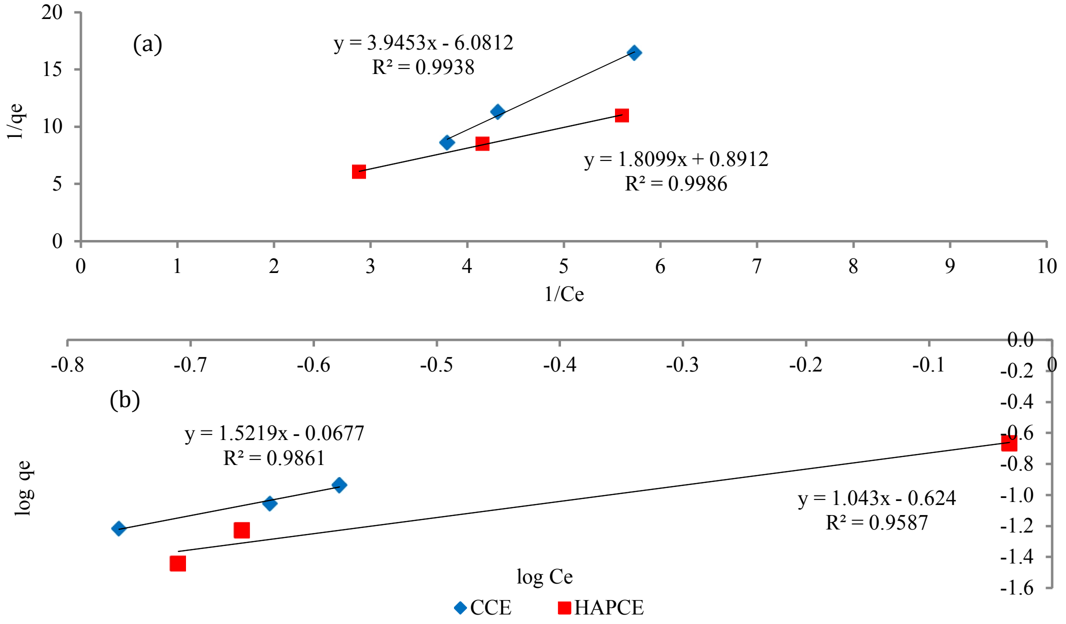

Fig. 4: Isotherm model for CCE and HAPCE (a) Langmuir (b) Freundlich

the value of R_L within the range, $0 < R_L < 1$ that indicates it is well fitted with Langmuir isotherm model with R^2 is 0.9938 and R^2 is 0.9986, respectively, as illustrated in Fig. 4(a) and as a comparison to Freundlich isotherm model in Fig. 4(b). The monolayer adsorption mechanism was found in Langmuir and heterogenous layer in Freundlich isotherm. Other than that, Freundlich isotherm model results indicate of sorbent-adsorbate is cooperative adsorption. Meanwhile the unfavorable sorption of iron was obtained (n = 0.6667) as shown in Table 2. Other than that, the HAPCE adsorbent, shown the normal process with n=1.230 that indicates the favorable adsorption which is fitted with Freundlich isotherm.Thus, both of CCE and HAPCE bio-sorbent were best fitted on Langmuir isotherm as both value of R^2 obtained in Langmuir Isotherm much higher rather than the R^2 value in Freundlich Isotherm (Fig. 4(a) and Fig. 4(b) as well as Table 2).

Table 2: Langmuir and Freundlich isotherm models for both bio-sorbents

Metal ion (Fe)	Langmuir			Freundlich		
	K_L (L/mg)	R_L	R^2	n	Log K_F	R^2
CCE	-925.9000	0.0005397	0.9938	0.6667	-0.0677	0.9861
HAPCE	146.2146	0.00341	0.9986	1.2301	-0.6240	0.9587

CONCLUSION

The adsorption of Fe ion onto CCE and HAPCE were depended on the two variables, which are contact time and bio-sorbent dosages. Percentage removals in this experiment differ depending to increase of contact time and dosage of bio-sorbents used whereby each parameter give different results depending on the bio-sorbent material. Final concentration of aqueous solution is prominent aspect that needs to comply with WHO drinking water guidelines, which is less than 0.3 mg/L of Fe ion in drinking water and 1.0 mg/L in natural river water. Both CCE and HAPCE has been proved to have a potential in Fe ion removal whereby the final treated concentration is below than the guideline, 0.1635 mg/L and 0.1785 mg/L, respectively. The removal percentage of CCE and HAPCE bio-sorbent increased up to 91.83% and 91.07% with the initial concentration of 2.0 mg/L which were successfully removed by using 0.30 g of CCE at equilibrium contact time of 50 minutes and less 10 minutes of 0.60 g of HAPCE bio-sorbent dosage were obtained at constant of pH temperature and agitation rate in orbital shaker. In term of adsorption isotherms, both of bio-sorbent better being described in Langmuir isotherm model with correlation coefficient, R^2 is 0.9938 and 0.9986 compared with R^2 is 0.9861 and 0.9587, respectively of Freundlich isotherm model. The selected method in this study is superior to previous studies with different selection of targeted final concentration which could be complied with drinking water quality guideline by using of eggshell waste. Overall, it can be concluded that commercialized chicken eggshell and hybrid *Akar Putra* chicken eggshell both are capable in removal of iron ions from the aqueous solution. Both are potentially applicable in the drinking water treatment approach. However, CCE can be considered as the best bio-sorbent material compared to HAPCE providing an economical and efficient sounds portable water treatment approach option especially for drinking water supply.

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CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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AUTHOR (S) BIOSKETCHES

Nasir, H.M., Ph.D. Candidate, Department of Environmental Sciences, Faculty of Environmental Studies,Universiti Putra Malaysia, 43400 UPM Serdang, Selangor Malaysia. Email: *hanisah.nasir@gmail.com*

Azmi, A., BSc., Department of Environmental Sciences, Faculty of Environmental Studies,Universiti Putra Malaysia, 43400 UPM Serdang, Selangor Malaysia. Email: *aqilahazmi@gmail.com*

Aris, A.Z., Ph.D., Associate Professor, Faculty of Environmental Studies, Universiti Putra Malaysia 43400 UPM Serdang Selangor, Malaysia and Laboratory of Halal Science Research, Halal Products Research Institute, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia. Email: *zaharin@upm.edu.my*

Praveena, S.M., Ph.D., Assistant Professor, Department of Environmental and Occupational Health, Faculty of Medicine and Health Sciences, Universiti Putra Malaysia 43400 UPM Serdang, Selangor Darul Ehsan, Malaysia. Email: *smpraveena@gmail.com*

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