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Biosorption of heavy metals: a case study using potato peel waste

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ABSTRACT

Potato peel waste (PPW) from food processing was used for removing As^{3+}, Pb^{2+}, and Hg^{2+} heavy metals from water. The response surface methodology (RSM) and the central composite design (CCD) were employed for determining optimal conditions for heavy metal removal. The statistical analysis indicates that the effect of pH is the most significant parameter. The optimal condition for achieving the maximum removal was obtained for removing different metals using RSM. Desorption study indicates its good reusability within three recycling steps.

Keywords: Biosorption; Central composite design; Response surface methodology; Heavy metal

1. Introduction

The rapid development in new renewable energy i.e. solar power generation-electricity storage system and information technology of touch screen manufacturing relies heavily on metallurgy of critical metals i.e. rare earth, lithium, and cobalt processing, which has posed the big demands for cost-effectively and environmental-friendly approaches in removing toxic heavy metals that were liberated during metallurgical process [1–4]. The conventional processing approaches i.e. chemical precipitation [5], electrolytic recovery [6], ion-exchanges [7], solvent extraction [8], and adsorption [9–12] appear to be more competitive in processing wastewater with relatively high concentrations of target pollutants. For a relative low concentration of heavy metals in contaminated wastewater, biosorption by biomass such as agricultural wastes is one of the best candidates for cost-effective and economical concentrating the heavy metals from solution [13–15]. Prior arts have shown that the rich surface functional groups on the biomass, which creates metal binding *via* different mechanisms, facilitates the metal ions removal [16,17]. As potato is the world's the fourth favorable vegetable and is widely planted in Australia [18]. The annual potato production in Australia is approximately 1.5 million tonnes, which creates over 100,000 tonnes of PPW during food processing [19]. This creates big potential and opportunities for industrial scale utilization, reuse and high value conversion of these renewable resources, which is generated from food processing industry [20,21]. The application of food processing waste is extensively explored during the last decades. The efforts of converting them into energy, chemicals and adsorbents, have been extensively trialed [22]. Among these approaches, preparation of adsorbent is still regarded as one of the most practical and economical approaches for resources reuse and high-value conversion [23].

The RSM is a set of mathematical and statistical techniques seeking to optimize an objective function that is affected by multiple factors using the design of experiments (DoE) methods and statistical analysis. Instead of seeking the optimal solution within a large number of randomly generated candidates, RSM utilizes the reduced and simplified experimental designs to gain a thorough understanding

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of the system as well as to obtain the optimal combination of operating parameters [24]. Because of these advantages, it has been widely applied in the optimization works. Since biosorption is a complex process, of which involves many factors that will contribute to the final biosorption capacity, it will be very helpful in elucidating the impact of each individual process parameter and their combination effects upon the heavy metal removal response. This initiates our research for statistical study of biosorption of heavy metal using PPW. Up to date, the statistical optimization study of biosorption for heavy metals using PPW in order to find out the impact of each individual process parameter and their combination upon the heavy metal removal response, to the best of our knowledge, has rarely been published before. Therefore, in this work, the effects of process parameters such as pH value, adsorption temperature and duration using PPW in adsorbing As^{3+}, Pb^{2+}, and Hg^{2+} metal ions were extensively investigated.

2. Materials and methods

2.1. Biosorbent preparation

The inedible PPW collected from local market were dried in the oven at 60°C for 24 h. Then the dried PPW was crashed and sieved within a range of 0.05–0.45 mm to avoid transport limitations during batch adsorption.

2.2. Characterization of PPW and wastewater

Fourier transformed infrared spectroscopy (FTIR): The spectrum (Perkin Elmer Spectrum 2 with UATR-single reflection diamond) was used to study functional groups of samples. The sample was scanned in spectra range of 4000–370 cm^{-1}.

NCA: nitrogen and carbon element analyses was conducted in a Flash EA 1112 (Thermo Scientific) elemental analyser to analyze the sample which was decomposed at 950°C with helium as carrier gas.

Elemental analysis was analyzed by using induced coupled plasma-optical emission spectroscopy (ICP-OES) (OPTIMA 7100DV, Perkin Elmer, USA) with the aid of microwave, detailed sample digestion procedures could be found in prior reports [25].

The content of lignin, cellulose, and hemicellulose in PPW was analyzed by standard Van Soest and Klason lignin analysis method [26,27].

2.3. Experimental design and statistical analysis

RSM is a set of mathematical and statistical techniques seeking to optimize an objective function that is affected by multiple factors using the design of experiments (DoE) methods and statistical analysis [28]. Instead of seeking the optimal solution within a large number of randomly generated candidates, RSM utilizes the reduced and simplified experimental designs to gain a thorough understanding of the system as well as obtain the optimal combination of operating parameters. A CCD with three independent variables in this work was investigated to study the response pattern and to determine the optimal combination of pH, adsorption temperature, and duration to maximize metal ions removal. The design with three independent variables at five different levels (total 17 runs) was adopted to find offset, linear, quadratic and interaction terms using the following equation [29]:

$$Y = b_0 + \sum_{i=1}^{3} b_i X_i + \sum_{i=1}^{3} b_{ii} X_i^2 + \sum_{i<j,j=2}^{3} b_{ij} X_i X_j \quad (1)$$

The range and levels of variables are shown in Table 1. The statistical significance of regression term was checked by analysis of variance, ANOVA. In this work, each individual heavy metal removal rate was set as the optimization goal. The samples were set as the following patterns: PPW-p1-p2-p3-x, where p1 represents pH value, p2 represents temperature, and p3 represents duration during adsorption, and x represents specific heavy metal ion. For example, PPW-3-50-20-As represents adsorption was performed at pH value of 3, at 50°C, with duration of 20 min for adsorbing As^{3+}.

2.4. Biosorption experiment

The concentration of stock solution contains each heavy metal was 1000 mg/L. Then the stock solution was diluted with ultrapure water (MilliPore Milli-Q) to specific concentrations for equilibrium and kinetic adsorption studies. Ammonia solution and hydrochloric acid were used to adjust pH for adsorption and desorption test. For CCD studies, the initial 50 mg/L concentration of different metals was prepared for optimization study. During test, the aqueous solution (100 mL) and 100 mg PPW were placed in the vessel on a shaker with setting at 200 rmp in flask (250 ml), which was then immersed in a water bath at different temperatures, 5-mL aqueous samples were taken from the solution at different times to be analyzed using ICP-OES. The amount of adsorption q_t (mg/g) at the time of t was calculated using the equation [26]:

$$q_t = \frac{(C_0 - C_t)V}{W} \quad (2)$$

where C_0 and C_t are the liquid-phase concentrations of heavy metals ion solution at initial and time t (mg/L), respectively. V is the volume of the heavy metal solution (l), and W is the mass of the dry adsorbent used (g).

In order to obtain the isotherm of the adsorbing different metal ions, the experiments were conducted to determine the equilibrium. The results indicated that the time required to reach equilibrium was 24 h for PPW, therefore we adopted 24 h as the time for metal ions adsorption to reach equilibrium. The adsorption equilibrium experiment

Table 1
Range and levels of independent process variables used for CCD

Independent variables	Symbols	–ß	–1	0	1	ß
pH	X_1	3	4	5	6	7
Temperature, °C	X_2	30	40	50	60	70
Treatment duration, min	X_3	10	50	90	130	170

was carried out as follows: the initial concentration of metal ions varied from 10–50 mg/L in a 250 mL flask loading with 100 mg PPW. The solution was then shaken in a water bath at 25°C for 24 h. The resulting concentration of different metal ions in the aqueous phase after equilibration was determined by ICP-OES using the followings:

$$q_e = \frac{(C_0 - C_e)V}{W} \quad (3)$$

where C_0 and C_e are the liquid-phase concentrations of the metal ions solution initially and at equilibrium (mg/g), respectively. V is the volume of the solution (l), and W is the mass of the dry adsorbent used (g).

Reversibility studies were conducted as the following: the adsorptions was first conducted using fresh PPW for 24 h. After adsorption, the saturated PPW was put in 100 ml of 5% hydrochloric acid for one hour at 25°C in a 250 ml flask to recover PPW. The filtrate from desorption was analyzed by ICP-OES. The biosorption-desorption cycles of removing different heavy metals were repeated three times in order to determine the reversibility of biosorption.

3. Results and discussion

3.1. Property of PPW

The compositional and elemental analysis of PPW conducted by NCA and ICP-OES, are shown in Table 2. Three main components of lignin, cellulose, and hemicellulose are the main compositions in PPW. The fraction of lignin component in PPW is very close to the straw derived biomass but is much lower than that of timber derived biomass. The carbon element is the main element followed by oxygen, hydrogen, and nitrogen, indicating its carbohydrate property. The calcium metal element was detected by ICP-OES using microwave digestion method, while the heavy metal elements used in this work was in non-detectable level by ICP-OES analysis.

The FT-IR spectra comparison between PPW and PPW absorbed with different heavy metals is shown in Fig. 1a. The characteristic peaks for carbohydrates, lignin, and hemicellulose in PPW were observed in spectra of PPW and PPW adsorbed with different metals. These appreciable peaks are in 3250 cm^{-1} (referring to out of plane stretching H–O), 2910 cm^{-1} and 2850 cm^{-1} (referring to C–H groups from carbohydrates), 1580 cm^{-1} (characteristic C=O stretching), 1070 cm^{-1} (characteristic C–O stretching of carbohydrate substance), 1500 cm^{-1} and 1750 cm^{-1} (referring to C–O phenolic, carboxylic, and alcoholic groups), 1080 cm^{-1} (characteristic C–O stretching of carbohydrate substance), 870 cm^{-1} (characteristic adsorption peaks of the valence vibration of CH groups in lignin) [30] and 760 cm^{-1} (the valence vibration of C–O bond, and deformation vibrations of C–H groups in lignin and hemicellulose) [27,31]. The spectrum of PPW indicates

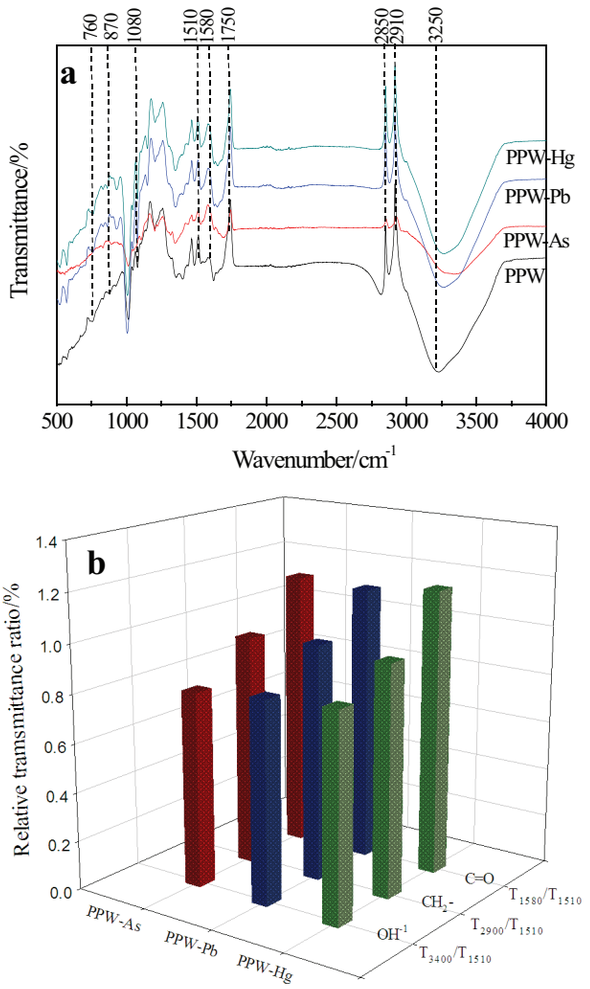

Fig. 1. a) FTIR spectrum of PPW, PPW-As, PPW-Pb and PPW-Hg, where adsorption was conducted at the optimal condition from RSM, b) relative transmittance ratio comparison of PPW absorbing different metals.

Table 2
Compositional and elemental analysis of PPW

PPW	Percentage (wt%)	NCA/ Composite	Percentage (wt%)	ICP-OES/ Element	Percentage (wt%)
Lignin	15	C	44	Ca	0.5
Cellulose	39	H	6	Mg	0.3
Hemicellulose	10	N	0.5	Si	<0.1
Others	36	O (by difference)	49.5	Fe	0.2

that rich surface groups providing available metal binding sites for biosorption during metal ions removal.

For metal biosorption, the surface functional groups such as OH– (3300 cm^{-1}), CH_2– (2900 cm^{-1}), and C=O (1580 cm^{-1}) are representative groups for metal ion binding and play vital roles during metal ions removal. In this work, relative intensities of PPW absorbed with As^{3+}, Hg^{2+} and Pb^{2+} metal ions using transmittance intensity at a wavenumber of 1510 cm^{-1} as references were calculated and the detailed methodology was described in prior literature reports [32]. The results are shown in Fig. 1b, it indicates that PPW in absorbing metal with larger charges (As^{3+}) presenting a lower relative transmittance ratio for the functional groups of OH–, CH_2–, and C=O, respectively. The possible reason could be that more available sites are needed for binding arsenic metal ion during biosorption [33,34]. For Pb^{2+} and Hg^{2+} metals, no significant ratio differences of those above surface functional groups were observed.

3.2. Statistical analysis and optimization of biosorption conditions

The experimental results associated with the interaction between each independent variable for removing different heavy metals are shown in Table 3. In terms of As^{3+} removal, the removal rate was found to be between 20 to 96%, respectively. For Pb^{2+} removal, the removal rate was found to be between 40 to 95%, respectively. And for Hg^{2+} removal rate, the removal rate was found to be between 20 to 40%, respectively. Table 4 gives the ANOVA results for the respective response of each individual heavy metal ion. The regression of model with high '*F-statistics*' value and low '*P*' value indicate the significances of the obtained models [35]. For As^{3+} removal, it is found that the term X_1, and X_1^2 are significant, while other terms have less significance for a response. For both Pb^{2+} and Hg^{2+} metal ions, it is found that only the term X_1^2 is significant. The second degree polynomial was derived to represent the correlation between heavy metal removal rate and three independent process variables as the followings:

$$Y_{As} = 87 - 23.9X_1 - 24.9X_2 - 0.2X_3 - 19.6X_1X_2 - 19.6X_1X_3 - 10.8X_2X_3 - 49.5X_1^2 - 33.1X_2^2 - 11.5X_3^2 \quad (4)$$

$$Y_{Pb} = 88 - 3.4X_1 - 9.5X_2 - 7.2X_3 - 25.1X_1X_2 - 10.9X_1X_3 - 2.2X_2X_3 - 56.4X_1^2 - 8.9X_2^2 - 8.5X_3^2 \quad (5)$$

$$Y_{Hg} = 37 - 1.6X_1 - 3.7X_2 - 0.5X_3 - 7.8X_1X_2 - 6.1X_1X_3 - 3.7X_2X_3 - 19.7X_1^2 - 3.8X_2^2 - 0.2X_3^2 \quad (6)$$

The metal removal rate was evaluated *via* 3-D plots of responses as a function of two factors while keeping the third parameter being at the optimal condition (Fig. 2). For removal of heavy metals using PPW, response surface plots show that the removal of heavy metals (As^{3+}, Pb^{2+}, and Hg^{2+}) are minimal both at low and high variables levels; however, it is also observed a region where removal rate neither increases nor decreases, which indicates the existence of the optimal conditions for maximizing the heavy metal removal. The standard square root r^2 for the quadratic model was 0.94 for all heavy metals, which indicates over 94% of the variation in the response can be explained by the model. The obtained adjusted r^2 was close to the experimental r^2 and predicted r^2. The adequate precision (AP) is basically a measure of S/N (signal to noise ratio), it gives a factor by which one can judge the obtained model to see

Table 3
Three factor CCD with experimental results of dependent variables

Runs	Code values			As^{3+} removal %	Pb^{2+} removal %	Hg^{2+} removal %
	X_1	X_2	X_3			
1	0	0	–ß	94	91	38
2	–1	–1	+1	85	57	37
3	0	0	0	79	83	36
4	+1	–1	–1	66	73	32
5	+1	+1	–1	37	52	24
6	–ß	0	0	90	91	38
7	+ß	0	0	70	76	33
8	+1	–1	+1	46	59	27
9	–1	+1	+1	59	58	30
10	0	+ß	0	20	42	20
11	+1	+1	+1	33	49	23
12	–1	–1	–1	42	56	26
13	–1	+1	–1	86	88	37
14	0	0	+ß	96	85	40
15	0	–ß	0	92	40	20
16	0	0	–1	94	93	36
17	0	0	0	92	92	37
18	0	0	–ß	87	89	36
19	0	0	0	93	92	39
20	0	0	0	96	95	40

Table 4
ANOVA analysis for heavy metal removal with r^2 0.94, Adjust r^2 0.94, Predicted r^2 0.93, AP = 13

Source	DF	Prob>F As^{3+}	Prob>F Pb^{2+}	Prob>F Hg^{2+}
Model	9	0.0435	0.0378	0.0210
X_1	1	0.0228	0.6395	0.5136
X_2	1	0.0268	0.2489	0.1821
X_3	1	0.9794	0.2875	0.8235
X_1X_2	1	0.4903	0.2822	0.3222
X_1X_3	1	0.3759	0.5349	0.3224
X_2X_3	1	0.5976	0.8951	0.5073
X_1^2	1	0.0044	0.0004	0.0003
X_2^2	1	0.0363	0.4377	0.3311
X_3^2	1	0.3388	0.3768	0.9573
Residue	10	–	–	–
Lack of fit	6	0.0001	0.0005	0.0007
Pure error	4	–	–	–
Cor total	19	–	–	–

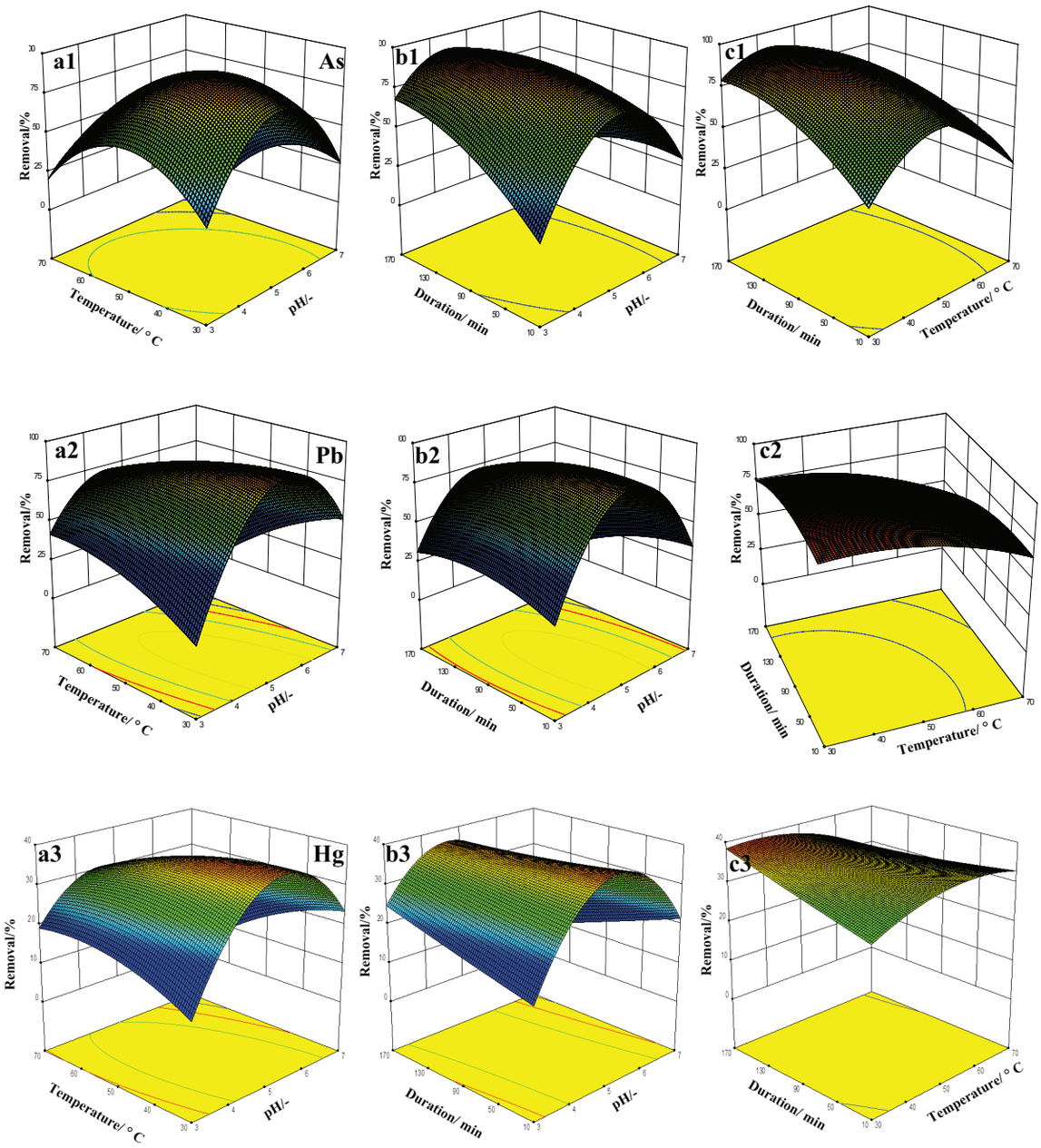

Fig. 2. Three-dimensional response surface for heavy metal removal a) As (III) removal response versus process parameters; b) Pb (II) removal response versus process parameters; c) Hg (II) removal response versus process parameters.

if it "adequate" to navigate through the design space and be able to predict the response. The desire values of AP should be over 4 [36]. In this work, the value of AP for all heavy metals removal is 13, indicating an adequate signal. The maximum heavy metal removal rate was set for optimization goal and 3 solutions were found for removing each individual heavy metal. For As^{3+} removal, the best condition obtained was PPW-5-50-30-As, which represented the arsenic ion was best removed by PPW through the following adsorption conditions: pH at 5, the temperature at 50°C, and adsorption duration in 30 min with removal rate reaching 96%. For Pb^{2+} removal, the best condition was PPW-5-30-20-Pb, which represented the lead ion was best removed by PPW through the following adsorption conditions: pH at 5, the temperature at 30°C, and adsorption duration in 20 min with removal rate reaching 94%. For Hg^{2+} removal, the best condition selected was PPW-5-30-50-Hg, which represented the mercury ion was best removed by PPW through the following adsorption conditions: pH at 5, the temperature at 30°C, and adsorption duration in 50 min with

removal rate reaching 38%. The additional experiments were conducted to further validate the model prediction by using the obtained optimal adsorption conditions. The heavy metal removal rate for the corresponding As^{3+}, Pb^{2+} and Hg^{2+} are 98%, 97%, and 40%, respectively. This indicates the experimental deviations for As^{3+}, Pb^{2+} and Hg^{2+} are 2%, 1% and 5%, respectively. These results indicate that the obtained models are reasonable and acceptable.

3.3. Equilibrium and kinetic adsorption

The isotherm for different heavy metal ions was conducted at obtained optimal conditions for pH and temperature with 24 h for equilibrium by varying initial concentration from 10 to 50 mg/L. The Langmuir equation was employed as the followings:

$$\frac{1}{q_e} = \frac{1}{(K_L q_m)C_e} + \frac{1}{q_m} \tag{7}$$

where q_e represents the adsorbed amount on adsorbent ($g \cdot kg^{-1}$), C_e is heavy metal concentration in liquid phase when adsorption is in equilibrium ($mg \cdot L^{-1}$), q_m represents the maximum monolayer adsorption amount by adsorbent ($g \cdot kg^{-1}$), K_L is Langmuir constant (–), which is indicative of the degree of affinity of adsorbent. The isotherm of adsorbing different metals at corresponding optimal conditions using PPW is shown in Fig. 3 and resultant parameters are shown in Table 5. As reflected from the affinity value of K_L for different metals, PPW shows the strongest affinity for the lead ion. The maximum monolayer adsorption capacity of PPW for adsorbing different metals also follows the order of As^{3+}<Hg^{2+}<Pb^{2+}. Many hypotheses have been proposed to explain the factors that contribute to the biosorption process. One of the possible factors could be the highest atomic weight of lead ion, which generates the higher momentum energy and increases probabilities of effective collisions between metal ions and surface functional groups on PPW [37]. In addition, electrical charges also play important role in the ion-exchange process during biosorption. Because of two possible oxidative states (+2 and +4) of lead ion in solution, it facilitates its sorption on the surface of PPW [38].

The adsorption kinetics is important data for sizing of adsorption column. For kinetic analysis of the uptake of different metals by PPW, the pseudo-second-order equation [39] was employed as follows:

$$\frac{t}{q_t} = \frac{1}{kq_e^2} + \frac{t}{q_e} \tag{8}$$

where t is adsorption time (min) and k is kinetic constant ($g \cdot mg^{-1} \cdot min^{-1}$). The effect of adsorption duration at the optimal conditions (pH and adsorption temperature) for different metals is shown in Fig. 4 and resultant parameters are shown in Table 5. From kinetic adsorption results, it shows that the major uptake happens during the first 30 min, indicating the biosorption is a fast process. In addition, by comparing this result with the CCD optimization study, the deviation between the obtained optimal condition of adsorption duration and kinetics for Hg^{2+} ion was observed. The optimal biosorption condition of adsorption duration for Hg^{2+}removal (PPW-5-30-50-Hg) from CCD is

Table 5
Langmuir adsorption and pseudo-second-order kinetic parameters of PPW at optimal conditions

Pollutant		Langmuir	
Metals	q_{max} (g/kg)	K_L	r^2
As	2.7	1.8	0.93
Pb	10.3	6.3	0.95
Hg	5.5	1.3	0.94
		Kinetic	
Metals	q_{max} (g/kg^{-1})	k ($g \cdot mg^{-1} \cdot min^{-1}$)	r^2
As	1.7	0.4	0.99
Pb	9.3	0.1	0.99
Hg	5.1	0.2	0.99

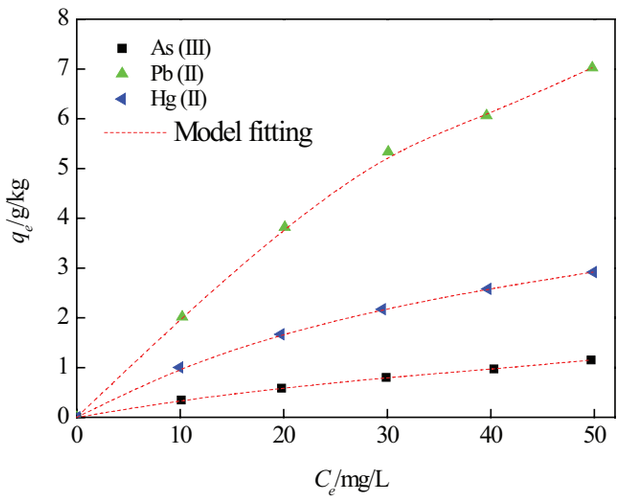

Fig. 3. Langmuir model for adsorbing metal ions by PPW.

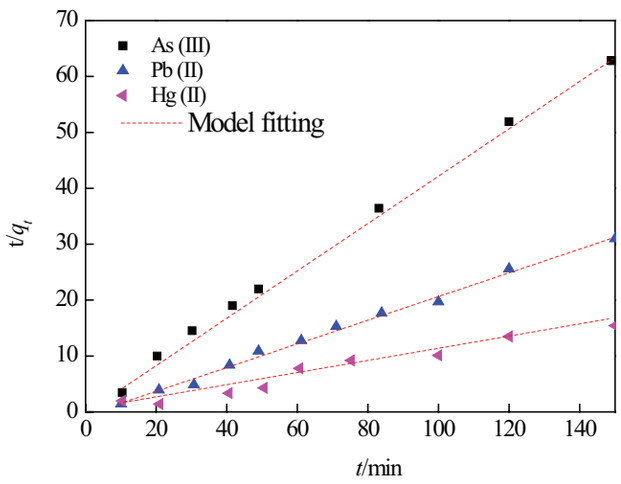

Fig. 4. Kinetic adsorption of metal ions by PPW using pseudo-second order kinetic model.

Table 6
Comparisons of heavy metal biosorption using different biomass, where CG represents coffee grounds, BB represents bacterial biomass, AG represents anammox granules, AB represents anaerobic biomass, SI represents Sargassum ilicifolium

Biomass	pH	q_{max} (g·kg^{-1})	Metals	Reference
CG	5.0	22.9	Pb	[40]
Hydrochar	5.1	5.5	Pb	[41]
SI	3.7	195	Pb	[42]
Biochar	5.0	47	Pb	[43]
BB	4.0	0.5	As	[44]
AB	5.0	0.2	As	[45]
Rice	5.0	0.2	As	[46]
Yeast	4.0	35	As	[47]
Leaves	5.5	37.2	Hg	[48]
Algae	6.0	42	Hg	[49]
Crab shell	6.0	8.3	Hg	[50]
AG	6.0	2.3	Hg	[51]
PPW	1.7	2.7	As	This work
PPW	9.3	10.3	Pb	This work
PPW	5.1	5.5	Hg	This work

50 min, while kinetics show that the major uptake occurs at 30 min. This discrepancy indicates that it takes longer times for PPW to adsorb Hg^{2+} ion when more process parameters were taken into consideration during optimization.

In Table 6, the comparisons of removing different heavy metals using different biomass for biosorption were made. Since there are many factors (experimental conditions) that contribute to the biosorption, many discrepancies still exist. The possible reasons that attribute to these adsorption removal capacity differences come from different physiochemical properties of the precursors such as specific surface area, charging of surface functional groups, electrolytic differences during adsorption, etc. Overall, our results are comparable to the results reported from prior literature reports, suggesting that the biosorption using PPW is a promising process in low level heavy metal pollution removal.

3.4. Reversibility study

The saturated adsorbed PPW was firstly recovered by adding acid to lower pH value in order to discharge the bonded metal ions. Then, the recovered PPW was used for next cycle of biosorption. This cycle was repeated for three times. The recovery of metal ions (as a percentage of adsorbed metal ion concentration) all fall within the range of 15% indicating at least 85% of active adsorption sites of PPW could be recovered by this pH swing regeneration process. The efficiency of reversibility of PPW in removing of different metals followed the order: Pb^{2+} (92.6%) >Hg^{2+} (88.2%)> As^{3+} (84.5%). The recovered PPW exhibit good biosorption capacity in Fig. 5 indicating its good reversibility. Therefore, heavy metals can be easily concentrated and recovered by this pH swing process and the resultant biodegradable PPW can be recovered and further reused for i.e.

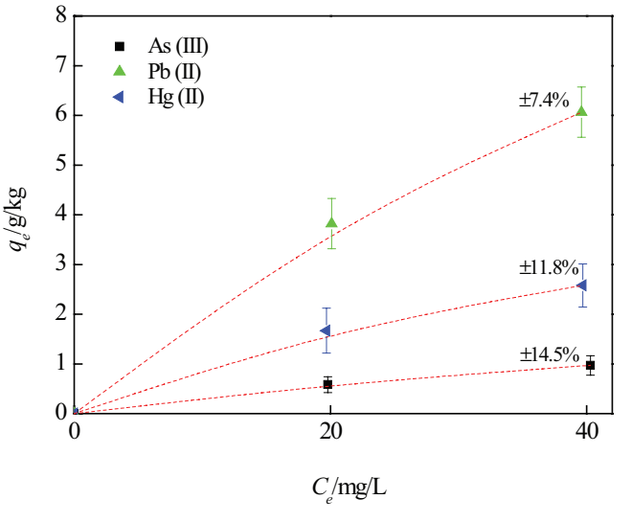

Fig. 5. Reversibility study of PPW with three round of adsorption and desorption.

soil conditioners, fertilizers without causing new environmental hazards by disposing.

4. Conclusions

The performance of biosorption using PPW shows great potential for cost-effective removal of metal ions in wastewater. The Langmuir model presents a good prediction of biosorption process for all metals. A lower relative transmittance ratio in adsorbing arsenic ion for the functional groups of OH–, CH_2–, and C=O, were observed from FTIR. The RSM and the CCD were successfully employed to optimize the process for biosorption of heavy metals using PPW. The maximum monolayer metal adsorption capacity obtained from Langmuir model, *e.g.* As^{3+} 2.7 (g·kg^{-1}), Pb^{2+}10.3 (g·kg^{-1}) and Hg^{2+} 5.5 (g·kg^{-1})were achieved. The pH value is found to be only significant to the metal uptake in ANOVA analysis. The reversibility study shows that over 85% of adsorption capacity of PPW could be recovered for all the testing metals over three round of recycling process.

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