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Sorption dominance of biowaste *Camelia sinensis* and *Gallus domesticus* to activated carbon, alumina, and silica

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ABSTRACT

Toxicity of heavy metals to higher life forms is ascribed to mobility and accumulation in water reservoir. Adsorption offers a promising alternative to treat wastewater. This article proposes green and economically viable options for the replacement of expensive commercial adsorbent. The spent *Camelia sinensis* (black tea) and *Gallus domesticus* (hen egg shell) powder under identical experimental conditions possesses better sorbing potential for cadmium (Cd) and lead (Pb). The adsorbents can be conveniently regenerated with HCl solution. The proposed adsorbents were found successful in situ remediation of textile, pharmaceutical, and leather industry effluents. Cost benefit and batch sorption analysis strongly recommends the present experimental endeavor for commercial scale up.

Keywords: Biowaste; *Camelia sinensis*; *Gallus domesticus*; Wastewater

1. Introduction

Environmental compartments are continuously being contaminated by industrial effluents. Solid and liquid waste mixes with groundwater and raises concentration of heavy metals higher than NEQS and WHO recommendations [1]. These metals serve no biological function; however, their presence in tissues reflects contact of the organism with its environment [2]. Industrial usage and commercial applications of Cd and Pb are common in fertilizer, foundries, petrochemicals, petroleum refining, and chlor alkali industries. The discharges of these metal work as cumulative poison, and added to the list of acknowledged endocrine disrupting chemicals [3]. The emission standards set by Pakistan Environmental Protection Agency for Cd and Pb defines concentration levels below 0.1 and 0.5 mg/L, respectively [4]. The irrational discharge calls for efficient methods for water purification and metal recovery. Biosorption technology has gained impetus integrity in and new low cost adsorbents are developed. The specific physical and chemical characteristics of adsorbents such as porosity, surface area, and physical strength make them a preferred choice. Benaissa [5] compared the abilities of four low-cost materials to remove Cd from aqueous solutions. However, exact mechanism of Biosorption is poorly understood. It becomes imperative to undertake intensive and extensive investigations for optimum output of bench scale research. The present study explores possibility of low cost novel adsorbents at two levels. Primarily, cost

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benefit analysis of biowaste (*Camelia sinensis* and *Gallus domesticus*) powder and commercial (Activated Carbon, Alumina, and Silica) materials as potential adsorbents is undertaken with identical experimental regime. Secondly, these adsorbents are applied in batch mode for the removal of Cd and Pb from synthetic metal salt solutions and actual industrial effluents.

Disposal of metal sorbed exhausted adsorbent creates another problem. This can be managed to some extent using elimination methods and Elution is considered as the most common elimination method [6]. Nitric acid, sodium citrate [7], HCl, H_2SO_4, H_3PO_4 [8], and EDTA [9] are extensively reported in literature for desorption studies. Another significant aspect of the present study is allowing recovery of metal ions and recycling of spent adsorbent for subsequent use.

2. Materials and methods

2.1. Biosorbent establishment

Biowaste samples were processed for the preparation of adsorbents following the protocol reported earlier [10,11]. The standard solution (1,000 mg/L) of Cd (II) and Pb(II) were prepared from cadmium sulfate ($CdSO_{4-8}/3H_2O$) and lead nitrate ($Pb(NO_3)_2$) procured from Merck. The concentration of metal ions in the sorption medium was determined using Flame Atomic Absorption Spectrophotometer (Varian AA-220, Australia). Cations uptake by adsorbents was calculated as percentage removal (%R) of metal ions by:

$$\%R = \left(\frac{C_i - C_t}{C_i}\right)100 \tag{1}$$

where C_i, and C_t (mg/L) are the liquid-phase concentrations of metal ions initially and at time t. To minimize contamination chances, glassware was treated with detergent water, followed by 10% nitric acid solution and finally rinsed with distilled water.

2.2. Protocol for batch adsorption

To assess the sorption efficiency, p^n factorial experiments are investigated to determine variables like induced sorbate concentration (C) and sorbent dose (M) on removal percentage of metal ions. The factor M and C is assessed at two levels (0.05 g/L; 0.1 g/L) and (25 mg/L; 50 mg/L). Levels are marked as 1 and 2 for lower and higher quantities, respectively (see Table 1). The interaction between factors (M and C) is identified by ANOVA table having 02 columns and 02 rows. The results are figured out from 04 cells, where each cell ($M \times C$) displayed N, $\sum X$, Mean, $\sum X^2$, Variance, Std. Dev., and Std. Err. The hypotheses are constructed on the basis of means and tested through independent estimate of the common variance. Factorial design experiments are performed for Cd and Pb removal separately by spent tea and hen egg shells under batch sorption protocol at 16˚C, described earlier [12]. Furthermore, same protocol is considered for metal removal from synthetic solutions by tested sorbents in comparison to commercial adsorbents.

Table 1
Two level factorial design

Trial	C_1, 25 mg/L	C_2, 50 mg/L
M_1, 0.05 g	C_1M_1	C_2M_1
M_2, 0.1 g	C_1M_2	C_2M_2

The sorption efficiency of biowaste is also rehearsed in industrial effluents sampled from different industrial area of Punjab, Pakistan. Each industrial aqueous sample is analyzed for its physiochemical parameters including pH, electrical conductivity, and background concentration of Pb and Cd. Metal adsorption from effluent samples is performed in raw and acid digested form.

2.3. Desorption protocol

The regeneration of biowaste and metal recovery is designed by employing desorbents of different chemical nature; deionized water (neutral); NaOH (basic); HCl & CH_3COOH (acidic) as preliminary tests. Metal sorbed biomass agitated against each of these stripping agents, and concentration of metal ions in the leachates is determined using FAAS. Amount of metal recovered (q_{td}) from the sorbent by desorption process at time t can be calculated by [13]:

$$q_{td} = \frac{q_e - C_t}{M_2} V_2 \tag{2}$$

where q_e is metal uptake capacity (mg/L) of biomass at adsorption equilibrium, C_t is the concentration of metal ions recovered at time t, M_2 is mass of loaded sorbent, and V_2 is volume of stripping agent.

3. Results and discussion

3.1. Multivariate data analysis

To determine the influence of C and M on percentage sorption of metal ions from aqueous medium with an interaction, multivariate analysis of variance (ANOVA) is executed. In the current investigation, main effects are the change of dose and concentration, and interaction of first order being intended for these two factors. To test the significance of factors at their levels, three different Null hypotheses are formulated:

Null hypothesis	Alternate hypothesis
$H_o{'}$: that *M*-means are equal, against	$H_1{'}$: Not all *M* means are equal
$H_o{''}$: that *C*-means are equal, against	$H_1{''}$: Not all *C*-means are equal
$H_o{'''}$: that there is no interaction, against	$H_1{'''}$: that interaction effects are not all equal to zero

These hypotheses are tested by comparing independent estimate of the common variance σ^2. For this purpose, total sum of squares is computed in the usual manner and the estimates of the variance are obtained by partitioning the total sum of squares into four components, corresponding to the four possible sources of variation. The variations are:

(1) Between dose (*M*).
(2) Between concentration (*C*).
(3) Interaction between dose (*M*) and concentration (*C*).
(4) Within cells about the cell means.

Significance level for all analysis is accepted at $\alpha = 0.05$ and the significance of all effects is tested at test statistic:

$$F = \frac{\text{estimated variance from “Between SS”}}{\text{estimated variance from “Error SS”}} \tag{3}$$

The sum of squares is computed for all the four combinations spent tea Cd (ST-Cd), spent tea Pb (ST-Pb), egg shells Cd (ES-Cd), and egg shells Pb (ES-Pb). Experimental runs were repeated twice and the ANOVA table generated (VassarStats: Website for Statistical Computation) are captured in Table 2.

3.1.1. Significance of main effects

Significance of first formulated hypothesis ($H_o{'}$: *M*-means are equal) is drawn from summary tables. Probability (*P*) values lead us to conclude rejection of this Null hypothesis for spent tea-Cd, egg shells-Cd, and egg shells-Pb combinations but acceptance for spent tea-Pb. Percentage removal expressed by main effects based on dose for first three combinations suggest that % sorption at low dose of sorbent is not equal to high dose. Statistical significance of these differences is computed by Tukey HSD test at 0.05 α level, compared with difference in marginal means of *M* effect marked as MμD, listed in Table 3. Results clearly indicate that MμD for sorption of Pb onto spent tea is lower than the critical mean difference (8.48).

P-value proposes rejection of second Null hypothesis ($H_o{''}$: *C*-means are equal). It means percentage removal of sorbate at its low concentration is not equivalent to higher one. Significance of difference verified through Tukey HSD tests leads to acceptance of alternative hypothesis, because all values of MμD are higher than its critical value.

3.1.2. Significance of interaction effects

Last hypothesis ($H_o{'''}$: there is no interaction) about interaction effects between sorbent dose and sorbate concentration is found significant only for Cd sorption by spent tea (ST-Cd). This conclusion is assumed on *P* and Tukey HSD test critical values. Significance is constructed through independent estimates of common variance σ^2, depicted in Table 4. Significant interaction between *M* and *C* is predicted through comparing variance σ^2 that are unique to four combinations. Variances at low level of *M* against both levels of *C* are observed as, M_1C_1 combination variance is in lower numbers than M_1C_2; similar pattern is reflected for M_2C_1 in judgment to M_2C_2. Alternatively, interaction of C_1 upon two levels of *M* (C_1M_1 and C_1M_2) has lesser variance than C_2M_1 and C_2M_2. Conclusive verdict extracted from interaction effects is that lower dose of spent tea is equally efficient to higher dose for removal of Pb. In addition, variance among data point is least for lower dosage ($C_1 = 0.05$ g) against lower induced metal ion concentration ($M_1 = 25$ mg/L) for all the four combinations of factors.

3.2. Effluents characterization

All thirteen industrial aqueous effluents are noticed to fall into two broad categories of pH, extremely acidic pH 2 is found for plastic and ordinance factory and basic range of 9–11 is depicted by others (see Table 5).

Highest value (258 μS/cm) of electrical conductivity is noted for sample collected from oil refinery

Table 2
ANOVA summary table of sorbent–sorbate interactions

	Spent tea Cd					Spent tea Pb				
Source	SS	df	MS	F	P	SS	df	MS	F	P
Between M	306.5	1	306.5	7.31	0.0192	224.83	1	224.83	3.7	0.0785
Between C	1,611.45	1	1,611.45	38.46	<0.0001	871.09	1	871.09	14.35	0.0026
Interaction	233.27	1	233.27	5.57	0.036	2.64	1	2.64	0.04	0.8448
Error	502.83	12	41.9			728.39	12	60.7		
Total	2,654.05	15				1,826.95	15			
	Egg shells Cd					Egg shells Pb				
Source	SS	df	MS	F	P	SS	df	MS	F	P
Between M	2,765.16	1	2,765.16	46.54	<0.0001	1,930.88	1	1,930.88	50.38	<0.0001
Between C	2,208.88	1	2,208.88	37.18	0.0001	1,032.74	1	1,032.74	26.95	0.0002
Interaction	164.89	1	164.89	2.78	0.1213	29.02	1	29.02	0.76	0.4004
Error	712.96	12	59.41			459.91	12	38.33		
Total	5,851.89	15				3,452.55	15			

Table 3
Evaluation of significance level for factors at their levels

	ST-Cd		ST-Pb		ES-Cd		ES-Pb	
Effects	HSD	MμD	HSD	MμD	HSD	MμD	HSD	MμD
Rows	7.05	15.27	8.48	7.49	8.39	26.29	6.74	21.97
Columns	7.05	20.07	8.48	14.75	8.39	23.49	6.74	16.06
Cells	13.6		16.37		16.2		13.01	

Table 4
Common variance σ^2 of treatments combination between ST-Cd

	C_1	C_2	Marginal means (M)
M_1	18.93	39.02	69.02
M_2	26.39	83.26	266.35
Marginal means (C)	19.78	129.16	

Table 5
Characterized parameters of real industrial effluents

				Background level (mg/L)	
#	Industrial effluents	pH	EC μS/cm	Cd	Pb
1	Brewery	5.13	0.787	−0.03	0.92
2	Ceramic	9.28	0.5	−0.08	1.08
3	Electroplating	9.07	1.1	−0.02	0.30
4	Glass	10.97	18.5	−0.08	0.96
5	Leather	7.15	33.9	0.00	1.95
6	Marble	9.23	21	−0.10	0.33
7	Oil mill	7.49	1.59	−0.05	0.77
8	Oil refinery	7.2	258	0.85	−3.16
9	Ordinance	2.36	5.05	0.17	−2.49
10	Pharmaceutical	7.89	2.45	−0.04	0.94
11	Plastic	2.12	3.24	0.52	0.06
12	Tanneries	9.54	1.1	−0.08	1.62
13	Textile	8.67	2.5	−0.04	1.08

indicating its pollution level, while least value of 0.5 μS/cm is observed for ceramic waste. Concentration of Pb and Cd is determined as background level; and almost all of the industrial units are strikingly releasing Pb above the permissible limit of 0.1 mg/L excluding oil mill, oil refinery, and plastic industry. Cd is noticed higher than limits only in plastic and oil refinery units. Analysis shows highest concentration of Pb in the leather effluent than the other samples, as reported in the literature [14].

3.3. Metal sorption from effluents

Comparative metal sorption by employing 0.05 g of both biowaste practiced in synthetic solutions revealed superior sorption of Pb than Cd for 20 min agitation time (Fig. 1). By keeping all the sorption variables constant, comparative study of toxic metals was repeated four times, depicted fondness of Pb for spent tea with 77% removal followed by 66% for Cd.

Same trend is observed for egg shells marking 15% higher sorption of Pb than Cd. In the same way, comparative study by Vimala and Das [15] reported higher removal of Pb than Cd for button mushroom in relationship to oyster and milky mushrooms. Pb removal from industrial effluents is opted principally due to its higher release level than permissible limit and secondly it's superior sorption by both biowaste from synthetic solution. Industrial effluents catering pH category acidic (plastic, ordinance), neutral (leather, oil refinery), and basic (oil mill, pharmaceutical, textile) are selected for biosorption technology.

Egg shells biosorption effectiveness is noted for effluents from textile and pharmaceutical industry while spent tea for leather industry (see Fig. 2). For other samples, background concentration of metal ion was below detection limit of the instrument. Higher Pb removal from effluents of textile and pharmaceutical industry by egg shells can be expressed as "like dissolves like." Egg shell powder was figured out as basic [11] by EDX analysis having 48% calcium content. Thus, preceded acidic nature spent tea at basic pH of pharmaceutical (7.89) and textile (8.67) industry effluents. Sorption of acid digested effluent samples is found insignificant.

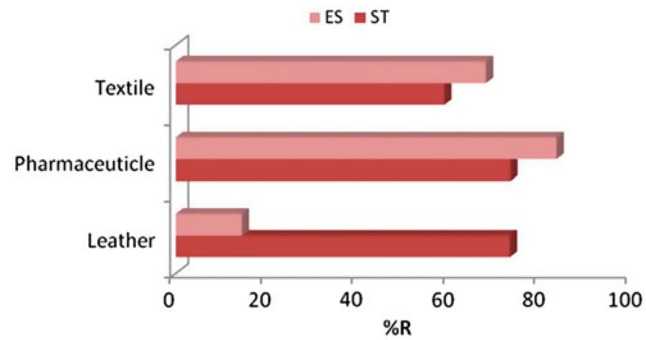

Fig. 2. Comparative sorption of spent tea and egg shells onto industrial effluents.

3.4. Sorption dominance of biomass

3.4.1. Comparative sorption efficacy for cadmium

Biowaste sorption potential against expensive adsorbents was analyzed at both levels of factor C and at higher level of factor M. Removal efficiency of 0.1 g mass of spent tea at C_1 (25 mg/L) within 20 min is found incredibly superior to commercially available expensive adsorbents (Fig. 3). Based on previous finding, the equilibrium contact time was accepted as 20 min for this study [16]. Higher removal by spent tea than silica, activated carbon, and alumina can be seen in order of 40, 26, and 20%. Similarly, Haris et al. [17] found 80% Cd sorption upon rice husks in comparison to 98% on activated carbon. To further verify feasibility of spent tea as economical adsorbent, removal at higher induced concentration (C_2) of Cd was also explored.

Fig. 4 narrates the same idea, and establishes supremacy of cellulosic waste over commercial commodities. Extension of agitation time beyond 20 min led to elevate 7% Cd removal, which proposes that unoccupied active sites of spent tea can sorb more toxic metal ions from solution by enhancing shaking time.

Egg shells, in comparison, are efficient equally to silica for Cd fixation at both levels of factor C, similar results were observed by Gutiérrez-Segura et al. [18] reported higher sorption of these two metals on carbonaceous material from pyrolyzed sewage sludge than activated carbon in column system.

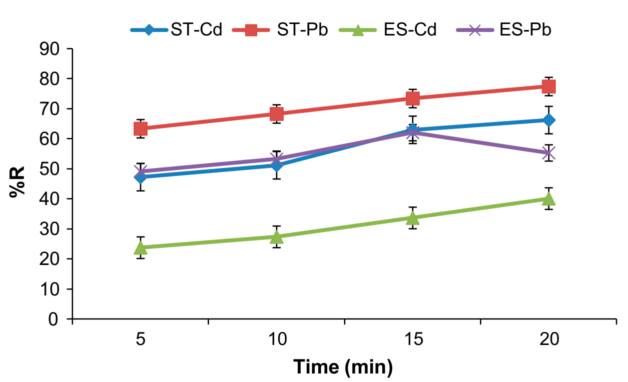

Fig. 1. Comparative sorption of spent tea and egg shells at 25 mg/L of metal ions concentration.

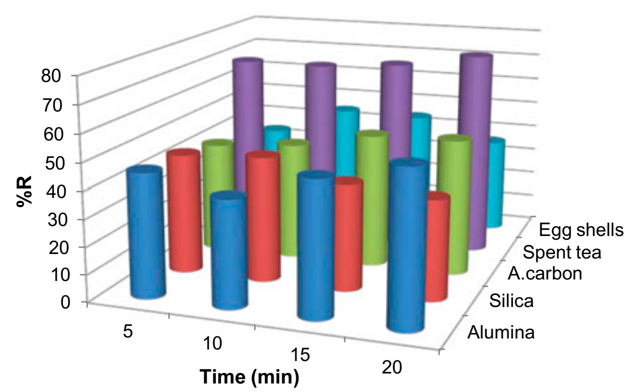

Fig. 3. Comparative sorption performance of biowaste for Cd at 25 mg/L.

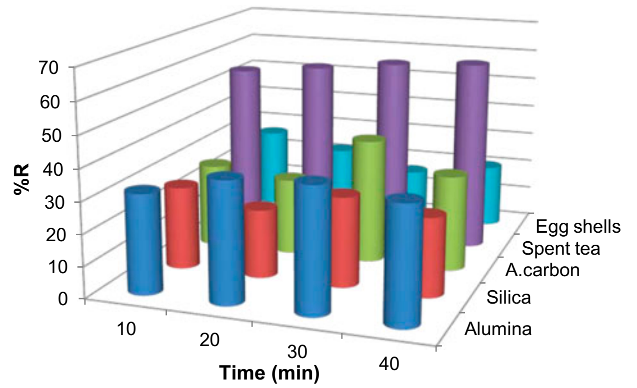

Fig. 4. Comparative sorption performance of biowaste for Cd at 50 mg/L.

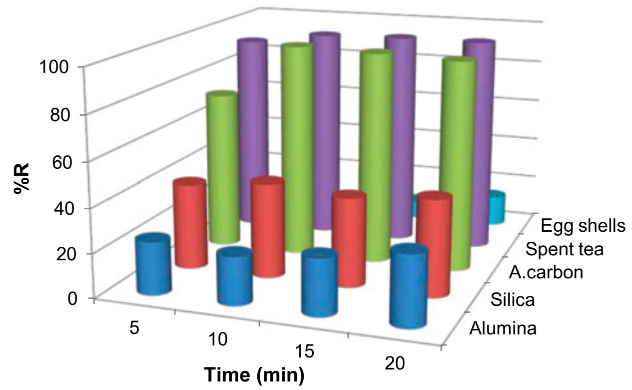

Fig. 5. Comparative sorption performance of biowaste for Pb at 50 mg/L.

3.4.2. Comparative sorption efficacy for lead

Comparative assessment of Pb removal from synthetic media is also explored under identical conditions. Strikingly, at lower solution concentration, spent tea and activated carbon sorbed Pb ions to such a periphery that cannot be measured as it was below detection limit (0.01 mg/L) of the instrument. However, this behavior escort spent tea and preferably at higher scale than Cd binding. Verification at enhanced concentration and time period is also executed, depicting metal removal trend as spent tea > activated carbon > alumina > silica > egg shells, supporting previous conclusion. Egg shells competing alumina for Pb removal instead of silica observed for Cd sorption (see Fig. 5). Partiality of Pb sorption over Cd can be professed by comparing hydrated ionic size. In solution, hydrated ion size is a dominant factor, smaller the hydrated ion higher is diffusion probability [19]. As hydrated ion size of Pb is lesser than Cd, suggest its sorption selectivity onto both adsorbents. Sorption dominance of spent tea leads to reasonably consider economical alternative to costly adsorbents.

3.5. Desorbent performance

Disposal of metal sorbed exhausted adsorbent creates another environmental pollution problem. This can be managed to some extent using elimination methods. Therefore, waste management through waste is executed with the perception of reusability after affirmation of their removal capacity. At present, metal release from sorbent surface is found highest by agitating sorbent with 0.05 M HCl solution followed by acetic acid solution of the same concentration. Similar ascendancy of HCl is reported for release of Cd than NaOH and H_2O using Nipa palm by Wankais et al. [13]. Mineral acid supremacy over organic acid is noted 16 and 34 folds for Cd and Pb discharge from spent tea in that order, while results are found comparable upon releasing metals bound onto egg shell surface. Greater desorption of both metal ions from spent tea surface in HCl solution was repeated four times and the trend can be attributed to weak sticking force explored during adsorption progression; physisorption on spent tea in comparison to chemisorption on egg shells. Fig. 6 clearly mark spent tea as superior sorbent than egg shells showing higher desorption potential. It is also noted that graph portrays higher desorption for metal release by spent tea similar to adsorption of metals from synthetic solution. This commands spent tea for the simultaneous charge and discharge in stripping media.

Concentration decay profile which is a ratio of metal concentration desorbed to adsorbed (q_{td}/q_a) is computed (see Fig. 7). The profile concludes that extent and rate of desorption is not strongly impacted on prolong time suggesting reusability of biomass.

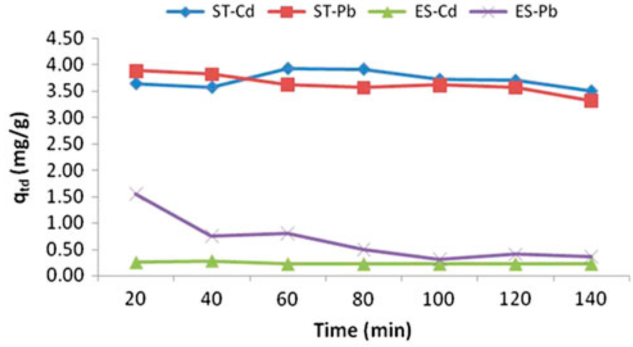

Fig. 6. Recovery of Cd and Pb from metal sorbed ST and ES biomass in HCl eluent.

Longer the contact time, higher is the probability of surface deterioration in desorption medium resulting lesser chance of regeneration.

3.6. Scale of selectivity and cost analysis

Factors influencing sorbent selectivity at industrial level include, low-cost, availability, profitability, ease of operation, regeneration, and above all environment friendly. Underutilized biomass, spent tea, and egg shells possess all these characteristics, and profitability is assessed through kinetic parameters and sorption mechanism discussed elsewhere [12]. The economics of biomass technical applications are improved by using discarded biomass instead of purposely produced biomass. Application of selected adsorbents for these highly toxic metals is verified in terms of cost with expensive commercial adsorbents. Cost (per kg) of activated carbon, alumina, and silica is 5,000, 2,000, and 1,600 PKR, respectively. Cost informations are seldom reported; hence, this contrast proposes these food residuals economical as well as accessible due to local availability.

The expense of individual sorbents varies depending on the degree of processing required. The total cost for the preparation of spent tea and egg shells as adsorbent is connected to its collection and minor processing steps like washing with water, drying in air feasible to maintain porous surface, because high temperature drying alter surface porosity. Benefit of using spent tea is also favorable due to no additional processing steps like crushing, homogenization, coning, quartering, and pulverization are needed for most adsorbents. Spent tea is accessible in powder or granular form, while preparatory step of egg shells is only crushing.

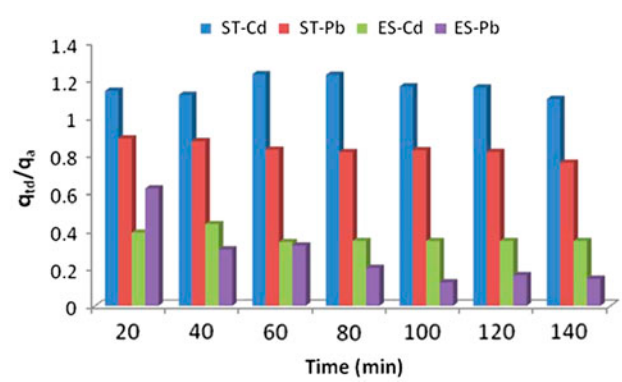

Fig. 7. Concentration decay profile of sorbents desorbed to metal sorbed.

4. Conclusions

Spent tea is noticed having huge appetite for Pb and Cd in comparison to activated carbon, while egg shells are found as productive as alumina and silica. The metal loaded spent tea and egg shells regeneration was explored at different pH and found best with mineral acid, hence metal free dried form could also be used as a fuel in boilers/incinerators; ash produced may be used for construction purpose like making fire bricks, leveling road side, or simply land filled. Safe disposal of metal-free waste entails two out of three R's that is reuse and recycle of natural resources. This approach not only ensures energy recovery, but at larger scale is conducive to environment. Adsorption as well as desorption trend is noted high for spent tea than egg shells. The complete removal of Pb from oil mill waste is achieved with spent tea. The results of this study are useful for managing the domestic waste and devising economical adsorbents for commercialization with no processing in the efficient removal of pollutants from wastewater.

5. Future recommendations

Water ways are the main receptor of untreated domestic sewage and industrial wastewater. Water quality is superficially executed as small research component with no integration. Therefore, coherent and comprehensive data generation for devising sound waste management plans is need of the day. Secondly, agro-based residues chiefly comprises almost same surface properties; thus, instead of exploring different adsorbents within same category need is to search remediation of broad range of toxic species from environmental media. As an attempt, egg shells sorption performance will be explored against acidic dyes and spent tea for phenol and nitrate.

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