



ADSORPTION OF COBALT AND ZINC FROM WASTEWATER BY NIGERIAN GRASS WASTE

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ABSTRACT

The Nigerian grass waste was used as an adsorbent for removing cobalt and zinc from the wastewater. The adsorption studies were conducted in single and binary solution. The waste was treated with sulphuric acid to enhance the properties like pore volume, particle size and surface area. The equilibrium data from the single component mode were checked with different models to investigate the adsorption mechanism. The equilibrium data were following Langmuir isotherm for cobalt and zinc. The adsorption in the binary solution mode showed synergism signifying that the addition of a component to a system increases the total adsorption capacity of the system.

Keywords: grass waste, cobalt, zinc, isotherm, interaction, synergism.

INTRODUCTION

Water is considered an essential source of life and energy [1]. The global world is faced with the problem of access to clean drinking water because of the rapidly growing industries and increasing population [1, 2]. Industries releases wastewater that contains organic, inorganic compounds, dyes and toxic metals [2]. Among these toxic metals are considered as one of the pollutants that are stable, non-degradable and cannot be broken down into simpler compounds by microorganisms present in the aquatic environment [3, 4]. Generally toxic elements such as zinc, cobalt, lead, mercury, chromium, copper and cadmium are released from the industries and causes harmful effect on human beings and the aquatic environment [5]. Thus the water pollution caused by these toxic elements is a major issue of environmental concern [6].

But importantly the industrial wastewater contains multiple components and each of these components interacts and competes with each other during the treatment process [7]. By investigating the synergistic or antagonistic behavior between the pollutants, the mobile nature of the pollutants in the wastewater system can be found. Therefore it is required to understand the behavior of the components in wastewater to improve the efficiency of the process [8]. Thus the present work studies the removal of cobalt and zinc from multicomponent wastewater.

Zinc which is one of the toxic metals is released from the wastewater of electroplating, refining and mining [9]. It has harmful effects such as nausea, fever, diarrhea, depression, restlessness and neurological symptoms [10]. Cobalt enters the aquatic life from the wastewater of nuclear power plants, mining, metal plating and petrochemical industries [10]. The exposure of human beings to cobalt results in damaging human blood cells and other diseases like leukemia and anemia [11].

Different treatment methods are used for removing the toxic elements from wastewater. The prominent among them are ion exchange, chemical precipitation, membrane process, biosorption, biodegradation and adsorption [12, 13]. Among these adsorption using is considered to be

efficient method for removing the pollutant from wastewater [14]. The process is easy in operation, different types of adsorbent can be used and the adsorbent can be regenerated [8].

Since the adsorbent prepared from non-renewable resource is expensive, it is required to prepare adsorbent from low cost and environmentally friendly agricultural waste material [15]. Therefore in the current work the adsorbent has been prepared from a grass weed Nigerian grass and used to treat zinc and cobalt from wastewater.

SIMULTANEOUS ADSORPTION AND THE INTERACTION BETWEEN THE POLLUTANTS

A binary solution having two components, interaction happens between the pollutants and the adsorbent. The interaction mechanism can be found by the comparison of the adsorption capacity of the i^{th} component in the solution having single component ($q_{i,m}$) and the adsorption capacity of the same pollutant in the binary mixture ($q_{i,b}$). The different types of possible interactions are various interactions are given as.

- Synergism: The adsorption capacity of the component increases when it is in association with the other component in the solution i.e. $q_{i,b} > q_{i,m}$.
- Antagonism: The adsorption decreases by the presence of other pollutant in the solution i.e. $q_{i,b} < q_{i,m}$.
- Non interaction effect: The adsorption is not affected by the other components i.e. $q_{i,b} = q_{i,m}$. [16, 17, 18, 19]



MATERIALS AND METHODS

Chemicals used

Cobalt nitrate hexahydrate (Finar Ltd) and Zinc sulphate heptahydrate (Finar Ltd) were used for the preparation of the solutions. The reagents sodium hydroxide pellets (Fischer Scientific Limited) and sulphuric acid (Merck India Ltd) were taken for the experiments.

Adsorbent preparation and the characterization

The Nigerian grass waste (*Pennisetum pedicellatum*) was obtained from the shrubs found near the larger trees in Manipal, Karnataka. It was first shred and washed with distilled water for 3-4 times to remove the impurities. It was then dried in a hot air oven at 120°C to remove all the moisture content and then made into fine powder [20]. The 10 g of dried powder was mixed with 40 ml of 0.2 M sulphuric acid. Then the slurry was kept in a muffle furnace at 350°C for 1 hr to improve the properties of material. Later the dried sample was washed with distilled water to neutralize the pH of the sample to 7 [21]. It was later dried for one day to remove the moisture content. It was reduced to a particle size of lesser than 0.075mm. Finally it was dried and used for the experiments.

The proximate analysis was conducted to determine the parameters moisture, ash content, volatile matter and fixed carbon [22]. The average particle size was measured with the help of particle size analyzer (CILAS 1064). The pore volume and surface area were estimated using BET equipment (Smart Instruments, India). The chemical bonds and functional groups present on the adsorbent surface were found by FTIR instrument, Shimadzu, Japan. The surface morphology of the adsorbent was investigated by Scanning Electron Microscopy (SEM), Zeiss Company, Germany. The presence of metal ions on the adsorbent were confirmed out using Energy dispersive X-ray (EDX).

Adsorption studies

The adsorption studies were carried out in 250 mL flask containing 200 mL of cobalt solution of 2, 4, 6, 8 and 10 mg/L concentration and 200 mL of zinc solution of 2, 5, 10, 15 and 20 mg/L in the single component solution. The experiments in binary mixture were conducted by taking

100 mL of cobalt solution and 100 mL of zinc solution of various concentration values as represented in Table-2. The effect of experimental conditions on the adsorption were found by the variation of pH from 4 to 8 and adsorbent dosage of 0.1 to 2 g. The optimum conditions of pH and dosage were determined as 7 and 2 g respectively. The equilibrium time was determined as 3.5 hours. The isotherm studies in the single solutions were carried out by stirring the solution with the adsorbent at pH 7. The solution was agitated in a shaker at 120 rpm speed, temperature of 298 K for 3.5 hrs. After the experiments, the concentration of cobalt and zinc were measured using atomic absorption spectrophotometer (Thermo Scientific, Australia) at a wavelength of 213.9 nm and 240.8 nm respectively.

RESULTS AND DISCUSSIONS

Adsorbent characterization

The pore volume and surface area and pore volume were measured as 250.13 m²g⁻¹ and 0.1391 cm³g⁻¹ respectively showing that capability of the adsorbent to remove the pollutant. The average particle size was found to be 54.11 μm and it shows that adsorbent has enhanced efficiency. The proximate analysis was carried to determine the moisture, ash, volatile matter and fixed carbon content and were found to be 5%, 31.5%, 21.5% and 42% respectively. The value of fixed carbon signifies that the adsorbent has better adsorption capacity for removing the adsorbate from wastewater [23].

FTIR analysis

The FTIR results of the adsorbent are represented in Figures 1 and 2. It was found that the peak obtained at 3608.81 and 3626.17 cm⁻¹ are because of the O-H bonding from the phenol group [24], the peak formed at 3487.30 and 3485.37 cm⁻¹ are due to the O-H stretching vibration band of hydroxyl group [25]. The peak at 1130.29 and 1103.28 cm⁻¹ exhibits C-N stretching [26] and the peak band at 1581.63 cm⁻¹ shows C=C stretching vibration [27]. The peak at 1710.86 cm⁻¹ were because of the stretching vibration from C=O bond because non-ionic carboxyl groups [28]. These bonds play a role in the adsorption of the pollutant on the adsorbent surface.

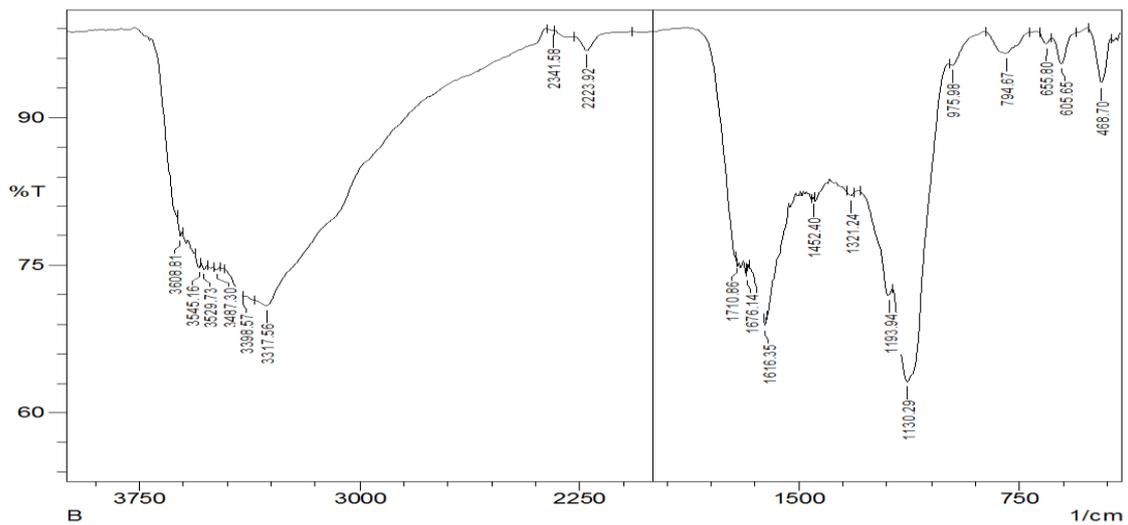


Figure-1. The FTIR analysis of the adsorbent before the adsorption.

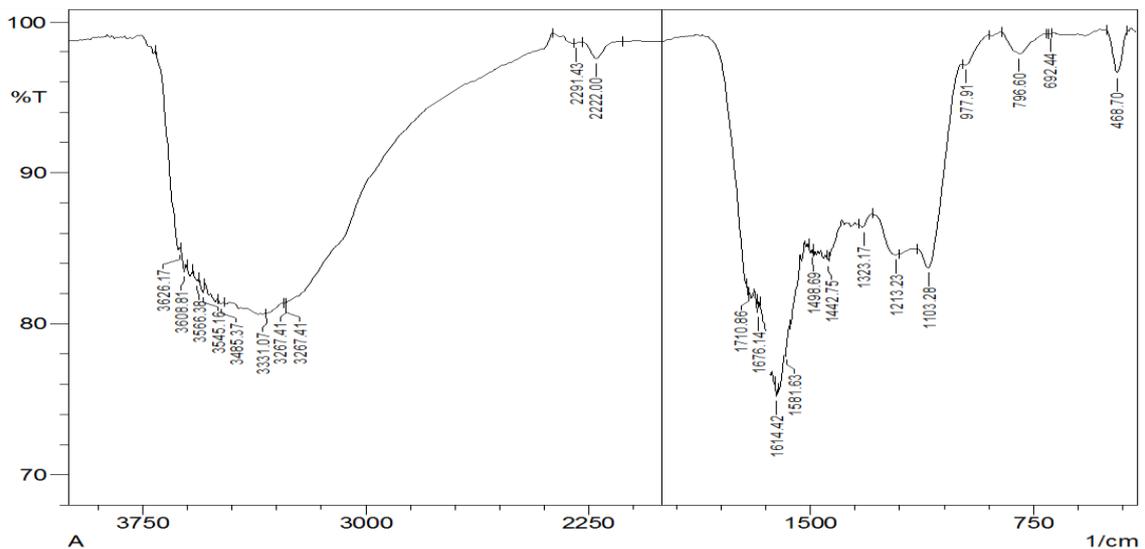


Figure-2. The FTIR analysis of the adsorbent after adsorption.

SEM and EDX analysis

SEM is a method employed for observing the surface structure of the adsorbent [29]. The surface morphology images of the adsorbent are shown in Figures 3 and 4. It can be analyzed from the SEM image that adsorbent surface is having porous structure and thus it provides required active sites for the adsorption process

[30]. It also signifies that the pores are found to be responsible for the accommodating the pollutant on the adsorbent surface [31]. It was also observed that after the adsorption, the adsorbent surface becomes rough. This is because of the reason that the pores are occupied by adsorbate molecules [32].

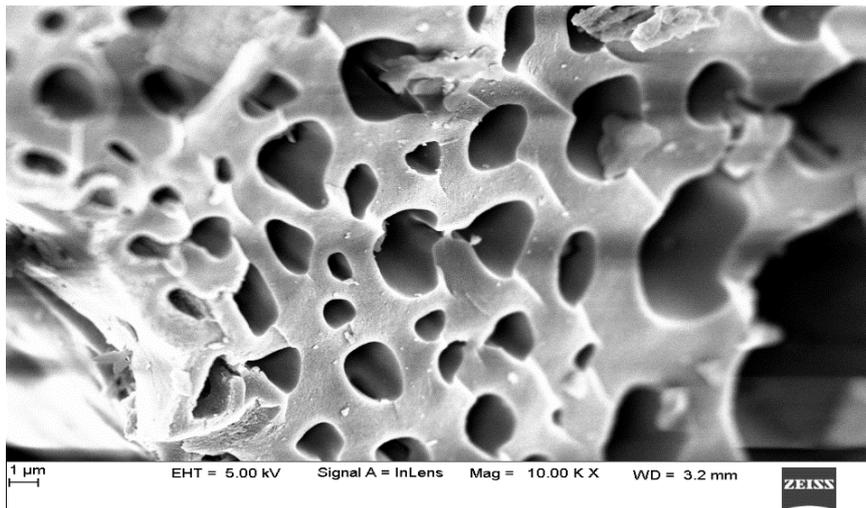


Figure-3. SEM image of the adsorbent before the adsorption.

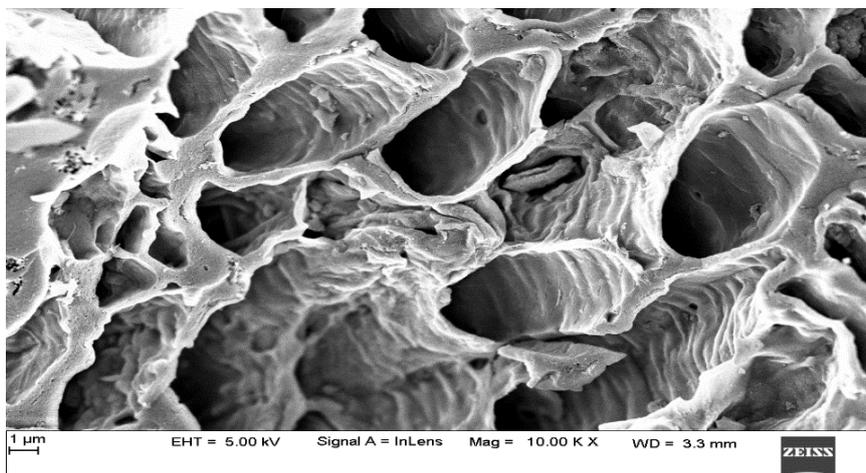


Figure-4. SEM image of the adsorbent after the adsorption.

Energy-dispersive X-ray spectroscopy (EDX) analysis was used to determine the chemical composition of the molecules on the adsorbent [33]. The EDX analysis of the adsorbents before and after the adsorbing the pollutant are shown in Figures 5 and 6. The EDX analysis before adsorption indicates the peaks for Ca^{2+} , K^+ , Mg^{2+} , Si which is because of the presence of plant nutrients in the grass waste [34]. It was observed that after the adsorption, peaks were obtained for metal ions Zn^{2+} and Co^{2+} on the surface of adsorbent. This shows the adsorption of metal ions on the surface through the ion exchange process [35, 36].

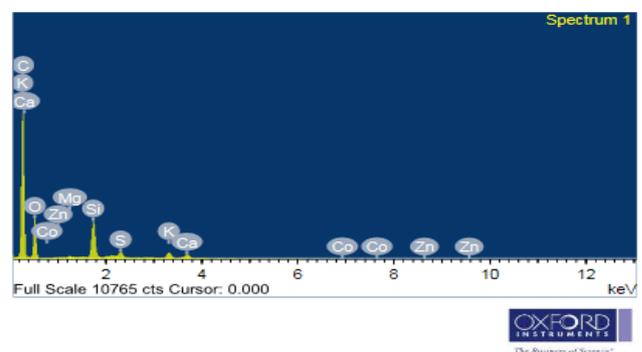


Figure-5. EDX analysis of the adsorbent before adsorption.

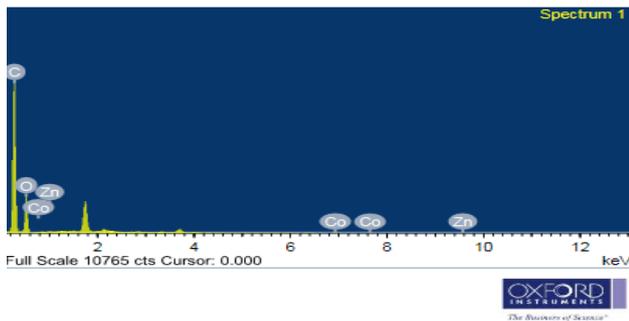


Figure-6. EDX analysis of the adsorbent after the adsorption

Isotherm studies.

Isotherm studies are important for analyzing and designing of adsorption separation process [37]. This is explained by establishing the equilibrium relation formed and the interactions between the solute molecules and the adsorbent [38]. Various models such as Langmuir, Freundlich, Temkin and Dubinin-Radushkevich (D-R) models were used for understanding the adsorption mechanism.

Langmuir isotherm is developed on the assumption that the surface of adsorbent is homogeneous with uniform active sites [39]. It also explains that homogeneous solid surface has the affinity towards the solute molecules resulting in adsorption in a single layer [40]. It also discusses that the sorption energy associated with each of the molecules is uniform [41]. It is given in the linearized form as

$$\frac{C_{eq}}{q_{eq}} = \frac{C_{eq}}{q_{mn}} + \frac{1}{k_{ln}q_{mn}} \quad (1)$$

k_{ln} ($L \text{ mg}^{-1}$) the affinity of the pollutant to the solid surface and q_{mn} (mg g^{-1}) the maximum adsorption capacity and q_{eq} (mg g^{-1}) and C_{eq} (mg L^{-1}) are the adsorption capacity and

pollutant concentration at equilibrium. The constants are obtained by plotting $1/q_{eq}$ against $1/C_{eq}$.

Freundlich isotherm is based on assumption that the adsorbent surface is heterogeneous, the solutes adsorbing on the surface increases with the concentration of solution [15, 42]. The surface sites will be occupied by many solute molecules and results in multilayer adsorption [43]. It is represented in the linear form

$$\log q_{eq} = \log k_{fn} + (1/n) \log C_{eq} \quad (2)$$

The model constants are evaluated from the plot of $\log C_{eq}$ v/s $\log q_{eq}$. The constant n is the adsorption intensity and k_{fn} the Freundlich constant.

Temkin isotherm explains that the adsorption energy decreases when the solute is adsorbed onto the solid surface and is because of the interaction happening between the solute molecules and the adsorbent [10, 44]. It is given as

$$q_{eq} = B_T \ln A_T + B_T \ln C_{eq} \quad (3)$$

and B_T the model constant which relates to heat of sorption and A_T the model constant. The constants are calculated by plotting q_{eq} v/s $\ln C_{eq}$.

D- R model is employed for determining the apparent free energy of adsorption and represented as [29, 44]

$$\ln q_{eq} = \ln q_{dr} + \beta \epsilon^2 \quad (4)$$

q_{dr} the adsorption capacity (mg/g), β the constant related to sorption energy and the constants are evaluated by plotting $\ln q_{eq}$ v/s ϵ^2 . ϵ the model constant is obtained from

$$\epsilon = RT \ln \left(1 + \frac{1}{C_{eq}} \right) \quad (5)$$

Table-1. The isotherm constants from various models for cobalt and zinc.

Component	Langmuir model			Freundlich model		
	$q_{mn}(\text{mg/g})$	k_{ln}	R^2	k_{fn}	n	R^2
Cobalt	1.3165	1.8736	0.9973	0.8231	1.4836	0.8128
Zinc	1.0708	1.6369	0.9326	0.7384	2.8008	0.6943
Component	Temkin model			D-R model		
	$A_T(\text{L/g})$	$B_T(\text{J/mol})$	R^2	$q_{dr}(\text{mg g}^{-1})$	β	R^2
Cobalt	4.1493	3.3573	0.6115	0.5710	7×10^{-8}	0.7468
Zinc	2.7497	1.1314	0.6582	0.4516	5×10^{-7}	0.668

The isotherm studies are conducted by varying the cobalt concentration in the range of 0 to 10 mg/L and zinc concentration from 0 to 20 mg/L. Various isotherm model equations were checked with equilibrium data obtained for single component solution. The model parameters calculated from the models and the regression coefficient

values for the components cobalt and zinc are given in Table-1. Based on the regression coefficient values, it is studied that the equilibrium data was fitting best with the Langmuir model which shows that the adsorption happens in monolayer and it is chemical in nature [45]. The larger the k_{ln} value for a component, the component will have



more affinity to the adsorbent. Thus it can be inferred that the cobalt is having higher affinity towards the adsorbent [46]. The adsorption intensity shows the favourability of the process. Then value higher than unity for both the components signifies that the process is favorable [43, 47]. The A_T value calculated for maximum for cobalt showing that the cobalt adsorption was more on the surface of the adsorbent [10].

The simultaneous adsorption of pollutants and the interaction effect in the binary solution

The isotherm studies were performed at different concentrations of cobalt and zinc. The adsorption capacity, percentage removal in the single and binary mixture and the interaction effect are represented in Table-2. The interaction effect was determined using additive rule. The comparison of total adsorption capacity of the pollutants in single solution and binary mixture were done. The adsorption capacity of the pollutants in the single

solution ($\sum_{j=1}^2 q_{j,m}$) and binary solution mode ($\sum_{j=1}^2 q_{j,b}$) are obtained as

$$\sum_{j=1}^2 q_{j,m} = q_{i,m} + q_{j,m} \quad (6)$$

$$\sum_{j=1}^2 q_{j,b} = q_{i,b} + q_{j,b} \quad (7)$$

If the value of $(\sum_{j=1}^2 q_{j,b}) > (\sum_{j=1}^2 q_{j,m})$ then synergism plays a significant role in the system. The value of $(\sum_{j=1}^2 q_{j,b}) < (\sum_{j=1}^2 q_{j,m})$ shows that antagonistic mechanism becomes the controlling factor [48].

The total percentage removal (*total rem%*) for the total and individual system was calculated as

$$total\ rem\% = \frac{(C_{o,i} - C_{e,i}) + (C_{o,j} - C_{e,j})}{(C_{o,i} + C_{o,j})} \quad (8)$$

where $C_{o,i}$ and $C_{e,i}$ are the initial and final concentrations for the components.

**Table-2.** The adsorption capacity and percentage removal of cobalt and zinc in single and binary component system.

$C_{o, cobalt}$ (mg/l)	$C_{o, zinc}$ (mg/l)	$q_{eq, cobalt}$ (mg/g)	$q_{eq, zinc}$ (mg/g)	$(\sum_{j=1}^2 q_{j,b})$ (mg/g) binary	$(\sum_{j=1}^2 q_{j,m})$ (mg/g) single	% rem, cobalt	% rem, zinc	(total rem%) , com	(total rem%) , ind
0	0	0	0	0		0	0	0	
2	0	0.1678	0	0.1678		83.9	0	83.9	
4	0	0.184	0	0.184		46	0	46	
6	0	0.4848	0	0.4848		80.8	0	80.8	
8	0	0.4147	0	0.4147		51.83	0	51.83	
10	0	0.8817	0	0.8817		88.17	0	88.17	
0	2	0	0.12	0.12		0	60	60	
2	2	0.1768	0.108	0.2848	0.2878	88.4	54	71.2	71.95
4	2	0.3819	0.1599	0.5418	0.304	95.47	79.95	90.3	50.66
6	2	0.5909	0.1437	0.7346	0.6048	98.48	71.85	91.825	75.6
8	2	0.7772	0.1172	0.8944	0.5347	97.15	58.6	89.44	53.47
10	5	0.9621	0.1322	1.0943	1.0017	96.21	66.1	91.19	83.475
0	5	0	0.4066	0.4066		0	81.32	81.32	
2	5	0.165	0.3944	0.5594	0.5744	82.5	78.88	79.91	82.05
4	5	0.3559	0.4018	0.7577	0.5906	88.9	80.36	84.18	65.62
6	5	0.5629	0.3625	0.9248	0.8914	93.71	72.5	84.07	81.03
8	5	0.7709	0.4209	1.1918	0.8213	96.36	84.18	91.67	63.17
10	5	0.9428	0.4145	1.3573	1.2833	94.28	82.9	90.48	85.88
0	10	0	0.8167	0.8167		0	81.67	81.67	
2	10	0.1502	0.691	0.8412	0.9845	75.1	69.1	70.1	82.04
4	10	0.383	0.8423	1.2253	1.0007	95.75	84.23	87.52	71.47
6	10	0.5734	0.8608	1.4342	1.3015	95.56	86.08	89.63	81.34
8	10	0.7486	0.8795	1.6281	1.2314	93.57	87.95	90.45	68.41
10	10	0.9426	0.8778	1.8204	1.6984	94.26	87.78	91.02	84.92
0	15	0	1.3193	1.3193		0	87.95	87.95	
2	15	0.197	1.3905	1.5875	1.4871	98.5	92.7	93.88	87.47
4	15	0.3738	1.3939	1.7677	1.5033	93.45	92.92	93.03	79.12
6	15	0.5888	1.4029	1.9917	1.8041	98.13	93.52	94.84	85.90
8	15	0.769	1.3879	2.1569	1.733	96.125	92.52	93.77	75.33
10	15	0.9181	1.3625	2.2806	2.201	91.81	90.83	91.22	88.04
0	20	0	1.7158	1.7158		0	85.79	85.79	
2	20	0.1672	1.7689	1.9361	1.8836	83.6	88.44	88.00	85.61
4	20	0.3878	1.8589	2.2467	1.8998	96.95	92.94	93.61	79.15
6	20	0.5773	1.8583	2.4365	2.2006	96.21	92.91	93.67	84.63
8	20	0.7666	1.8647	2.6313	2.1305	95.82	93.23	93.97	76.08
10	20	0.9302	1.8685	2.7987	2.5975	93.02	93.42	93.29	86.58

It was investigated from the Table-2 that the total adsorption capacity ($\sum_{j=1}^2 q_{j,b}$) for the binary solution was

higher than adsorption capacity ($\sum_{j=1}^2 q_{j,m}$) for the individual components. This shows that the system follows



synergistic type of behaviour. Similar nature of results was observed with total percentage removal in the combined and individual mode. The possible reasons are because of the solute properties such as ionic potential, ionic size, molecular polarity, affinity towards the adsorbent, molecular weight, and also the properties of the adsorbent like surface properties, porosity and functional groups [49, 50].

CONCLUSIONS

The current studies showed that the capability of the grass waste to adsorb cobalt and zinc from wastewater. The surface properties were found to be improved after the chemical treatment. The FTIR analysis showed the peaks which were responsible for adsorption and SEM analysis exhibited the adsorption of the pollutant on the adsorbent. The isotherm data fitted with Langmuir isotherm showing that the process follows chemical adsorption. The monolayer adsorption capacity was obtained as 1.3165 mg/g and 1.0708 mg/g for cobalt and zinc respectively. The total adsorption capacity of the binary solution was higher than that of single component solution demonstrating that the system follows synergistic behaviour.

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