

Treatment of gasifier based solid waste for preparation of useful sorbent and its application for lead removal

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Abstract: *In this research work, an attempt has been made to prepare useful sorbent from gasifier based solid waste, employing acid/base treatment. The main process parameters (such as acid concentration and temperature of pyrolysis) can affect the characteristics of the obtained activated carbon (expressed in terms of iodine number and methylene blue adsorption and specific surface area). Both treatments had a significant positive influence on sorption properties. The activated carbon presented a BET surface area of $715 \text{ m}^2\text{g}^{-1}$ and $558 \text{ m}^2\text{g}^{-1}$ for base and acid treatment respectively. The ability of for the removal of lead from aqueous solutions has been investigated. In the batch tests experimental parameters were studied, including solution pH, contact time and initial metal ions concentration. Maximum 96% removal of lead was observed. Removal of about 50–60% occurred in 20–30 min, and equilibrium was attained at around 60–80 min. The functional groups (C=O, S=O, –OH) present on the carbon surface may responsible for the adsorption of metal ion. The adsorption parameters were analysed using both the Freundlich and Langmuir models. The data are better fitted by the Langmuir isotherm as compared to Freundlich model. These experimental results indicated the potential use of gasifier based waste as a precursor in the activated carbon preparation process, thus representing an economically promising material.*

Keywords: activated carbon, chemical activation, sorption, lead

1. INTRODUCTION

Activated carbon is not only used as a adsorbent for the removal of dyes, but also it is used in food processing, pharmaceuticals, catalyst supports, solvent recovery, petroleum, mining, adsorption of radio nuclides, gasoline emission control, heavy metal recovery, contaminated air treatment due to its high adsorption capacity and high surface area [1]. In fact, it is limited to use because of its high price and high operation and regeneration costs. However, the demand for coal-based carbon has been growing interest in the all of countries [1, 2].

Precursors used for the production of activated carbons are organic materials that are rich in carbon, such as coal, lignite, and wood. Although coal is the most commonly used precursor, agricultural waste in certain

condition is a better choice [3]. Activated charcoal produced from residues would reduce the pressure on forests since wood is also commonly used for this purpose [4]. Many agricultural by-products such as coconut shell [4, 5, 6] grain sorghum [7], jackfruit peel waste [8] and Waste tea [9], have been discovered to be suitable precursors for activated carbon due to their high carbon and low ash contents. Agricultural wastes are considered to be a very important feedstock because of especially two facts: they are renewable sources and low cost materials [9].

Basically, there are two different processes for the preparation of activated carbon: physical and chemical activation. Physical activation involves carbonization of a carbonaceous precursor followed by activation of the resulting char in the presence of activating agents such as carbon dioxide or steam [10,3]. Chemical activation, on the other hand, is a single step method of preparation of active carbon. In this case carbonization of the precursor taken place in the presence of chemical agents. In physical activation, elimination of a large amount of internal carbon mass is necessary to obtain a well developed carbon structure, whereas in the chemical activation process all the chemical agents used are dehydrating agents that influence pyrolytic decomposition and inhibit formation of tar, thus enhancing the yield of carbon. The temperatures used in chemical activation are also lower than that used in the physical activation process, and therefore the development of a porous structure is better in the case of the chemical process [10,3]. The chemical agents used in the chemical process are normally alkali and alkaline earth metal containing substances and some acids such as KOH, ZnCl_2 , H_2SO_4 and H_3PO_4 . Considering limitations of conventional methods for metal removal, the most promising alternative appears to be the adsorption process [11]. Additionally; adsorption has been found to be superior to other techniques for water reuse in terms of the initial cost, simplicity of design, ease of operation and insensibility to toxic substances. Activated carbon (AC) is the most commonly used and most effective adsorbent. Nevertheless, its application fields are restricted due to its high cost [11]. This leads to search the new, cheap, and easily available and resource of carbon as a feedstock for activated carbon.

The adsorptive properties of activated carbon depend on the nature of the precursor, the type of activation (chemical or physical) as well as on the processing conditions. It has been shown that in case of chemical activation, concentration of the dehydrating

agent, impregnation ratio and pyrolysis temperature governs the properties of the resulting activated carbon [12]. Performances of the obtained carbons are generally expressed in terms of some properties, among which: surface area, adsorption efficiency towards iodine, and methylene blue are frequently considered [12, 13, 1, 14].

The removal of heavy metal contaminants from aqueous solutions is one of the most important environmental concerns because metals are biorefractory, and are toxic to many life forms [15,16]. Various sources of lead in water are battery manufacturing, basic steel, paper and pulp, metal plating, leather tanning, agrochemicals, petrochemicals and fertilizer industries. Higher concentrations of heavy metals in water and soils may increase the uptake of these metals by crops and potentially affect human health via food chains. Lead is ubiquitous in the environment and is hazardous at high levels. It is a general metabolic poison and enzyme inhibitor causes toxicity to the neuronal system, and affects the function of brain cells [17].

2. Materials and methods

2.1. Procedure of adsorbent preparation and activation

Gasifier based solid waste collected from (Arat electrochem Pvt. Ltd, Bapunagar), were crushed using a mechanical grinding machine and sieved to the selected particle sizes of 75-150 microns. After sieving and selecting, crushed particles were mixed with KOH / H₃PO₄ solution in a round bottom flask with condenser at 100°C and whole lab set up was properly adjusted and flow of water was maintained. in aqueous solution of either 0.5N-5N KOH and 5-15% phosphoric acid , for a period of 4-6 hours. Finally, the particles were thoroughly washed with water in a 2 litre beakers and filtrate for the cake formation; and dry using hot air oven to eliminate moisture.

2.2. Chemicals

All of the chemicals used were of analytical reagent grade. Deionised distilled water was used throughout the experimental studies. Stock lead solution (1000 ppm) was prepared by dissolving 1.599g of Pb(NO₃)₂, in 200 mL of DW, then 1.5 mL of conc. HNO₃ was added and diluted to 1000 mL with DW water. Working standards were prepared by progressive dilution of stock lead and zinc solutions using DW. HCl, NaOH solutions were used to adjust the solution pH.

2.3. Instrumentation

The following instruments were used to prepare, modify and characterize activated carbons as well as to conduct batch adsorption studies: mechanical grinding machine by sun instruments; muffle furnace (Lab Fine

manufacturers); hot air oven (Lab Fine manufacturers); BET surface area and Pore Size Analyzer (Nova 1000e); Fourier transform infrared spectrophotometer, FTIR (IRPrestige-21, Shimadzu, Japan); mechanical shaker (Sedko laboratory equipments); Digital pH meter (Systronics Pvt. Ltd.); Atomic absorption spectrophotometer (AAS-6300, Shimadzu, Japan); Ultraviolet—visible spectrophotometer (UV-1800, Shimadzu, Japan).

3. Characterization of the prepared carbon

Analysis of carbon samples show that the carbon is mainly in the form of very small crystallites with a graphite structure. To meet the engineering requirements of specific application, activated carbons are produced and classified as granular, powdered, or shaped products. Granular ACs are produced directly from granular precursors, such as sawdust and crushed and sized coconut char or coal [5]. The granular product is screened and sized for specific applications. Powdered-

Table. 1. Activated Carbon Analysis

Particulars	Units	CAC	GBC	GBC (CT)	GBC (TT)	GBC (CT+TT)
Ash	%	1.09	9.837	1.05	8.75	3.02
Moisture	%	3.2	4.85	4.1	3.87	3.8
Volatile organic matter	%	9.42	12.95	9.19	10.31	9.3
Carbon	%	86.3	72.32	85.64	77.02	83.86
Matters soluble in water	%	0.79	0.27	0.95	0.18	0.85
Hydrogen	%	2.58	3.88	2.59	1.43	2.45
Nitrogen	%	0.42	2.91	0.65	2.58	1.33
Sulphur	%	0.95	0.82	0.98	0.68	0.72
Oxygen	%	9.73	18.47	10.14	17.91	11.56
Specific surface area	m ² /gm	512	237	551	398	715
Conductivity	µs/cm	351	159	313	124	322

CAC= Commercially available carbon;

GBC= Gasifier Based Charcoal;

GBC(CT)=Gasifier Based Charcoal (Chemically Treated (1.5 N KOH));

GBC(TT)=Gasifier Based Charcoal (Thermally Treated);

GBC(CT+TT)=Gasifier Based Charcoal (Chemically Treated (1.5 N KOH) + Thermally Treated

-ACs are obtained by grinding granular products. Shaped AC products are generally produced as cylindrical pellets by extrusion of the precursor with a suitable binder before activation of precursor [2]. According to the International Union of Pure Applied Chemistry (IUPAC), the pores of a porous material are classified in three groups: micropores (width $d < 2\text{nm}$), mesopores ($2\text{nm} < d < 50\text{ nm}$) and macropores ($d > 50\text{ nm}$) [18].

1) Iodine number

The iodine number is defined in terms of the milligrams of iodine (I_2) adsorbed by 1 g of activated carbon when the iodine concentration is 0.1N. It is a measure of activity level (higher number indicates higher degree of activation), often reported in mg/g. The procedure of the iodine number determination is as follows: three dry samples of activated carbon are weighed out into three 250 ml conical flasks (sample

Table. 2. KOH activated GBC

0.5 N Solution (Iodine Number/Temp/hr)	1.5N Solution (Iodine Number/Temp/hr)	2.5N Solution (Iodine Number/Temp/hr)
1398/500°C /3	1423/500°C /3	1612/550°C /4
1479/650°C /3	1612/650°C /3	1977/750°C /4
1697/800°C /3	1784/800°C /3	2114/920°C /4

Table. 3. H_3PO_4 activated GBC

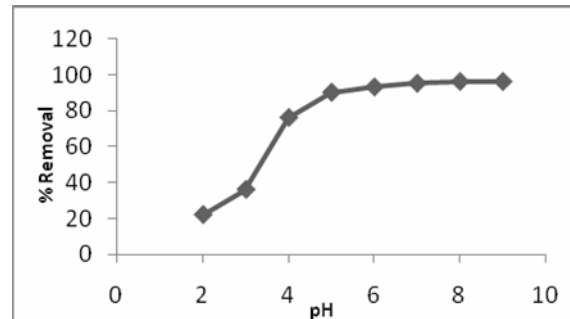
5% Solution (Iodine Number/Temp/hr)	10% Solution (Iodine Number/Temp/hr)	15% Solution (Iodine Number/Temp/hr)
1238/500°C /3	1411/500°C /3	1946/550°C /4
1328/650°C /3	1563/650°C /3	2215/750°C /4
1512/800°C /3	1649/800°C /3	2506/920°C /4

weight ranged between 300 and 600mg). 10ml of 5% (in weight) hydrochloric acid solution is added to each flask and then mixed until the carbon became wet. The mixtures are then boiled for 30 sec and finally cooling is done. 100ml of 0.05M standard iodine solution is added to each flask. The contents should vigorously shaken for 30 s and then immediately filtered. A 50 ml of each filtrate is titrate by a standardized 0.1N sodium thiosulfate solution. For each sample, the obtained iodine residual concentration should be included into 0.004 and 0.02 M. The plot of the amount of iodine fixed per gram of sorbent versus residual iodine concentration gives a straight line which allows determining graphically the iodine number [12].

2) Methylene blue (MB) adsorption

Some carbons have a mesopore structure which adsorbs medium size molecules, such as the dye - Methylene Blue. Methylene Blue adsorption is reported in g/100g (range 11-28 g/100g) [17]. Adsorption of methylene blue (MB) on activated carbon was carried out using a bath experiments method in a mechanical shaker. The effect of contact time, solution pH and adsorbent dosage were investigated. The adsorption process was carried out with two different initial concentrations at 100 ppm MB. In the pH study, the pH of MB solution was adjusted in the range of 2-9 by adding 0.1N hydrochloric acid or 0.1N sodium hydroxide. About 0.2gm adsorbent was then added to the solution and shaken at predetermined time.

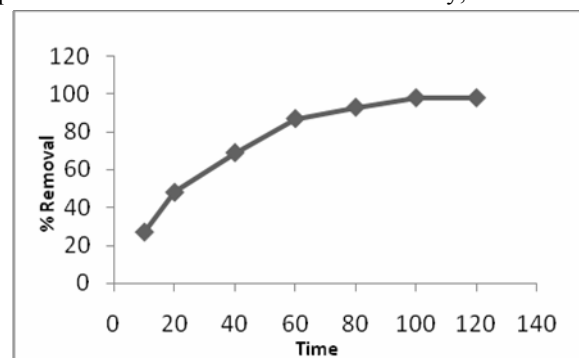
Fig. 1. Effect of pH on the adsorption(%) of methylene blue 100 ppm using activated carbon; Condition: contact time = 120



minutes, adsorbent dose=0.2gm/50ml

Fig. 2. Effect of adsorbent dosage(gm) on the adsorption(%) of methylene blue 100ppm using activated carbon Conditions: initial pH of MB solution = 6.5; contact time = 120 minutes

Adsorption tests were conducted by mixing 0.3 g of the prepared activated carbon with 100mL of 1000mg L^{-1} ethylene blue solution. After agitation during 24 h, the suspension measured at 660 nm, using an UV spectrophotometer. The effect of contact time was used to determine equilibrium time for the adsorption at optimized pH 7. For the effect of contact time study, 50 ml of MB



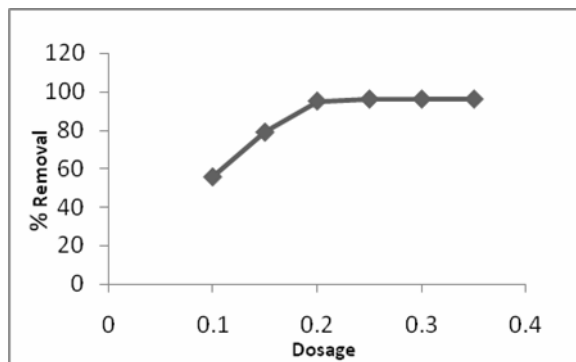
solution was added into a screw cap conical flask containing 0.20 g activated carbon and shaken constantly. Samples solutions were withdrawn at predetermined time intervals for the colour removal analysis.

The effect of adsorbent dosage on the removal of MB has been presented in Fig. 4. The amount of carbon required for quantitative removal of MB from 50 mL (50

Fig. 3. Effect of time on the adsorption(%) of methylene blue 100 ppm using activated carbon Conditions: initial pH of MB solution = 6.5; contact time = 120 minutes, adsorbent dose=0.2gm/50ml

mg/L) of solution was 150 mg. The percentage of removal increased to 99% with an adsorbent dose of 150 mg/50 ml.

It is apparent that by increasing the adsorbent dose, the removal efficiency increased but adsorption density, the amount adsorbed per unit mass, decreased. It is readily understood that the number of available adsorption sites



increased by increasing the adsorption dose. The decrease in adsorption density with increase in the adsorbent dose is mainly due to unsaturation of adsorption sites through the adsorption reaction.

3) Specific surface area

Surface area is one of the key indicators attributed to the adsorptive properties of porous materials. The most commonly used model in the determination of that parameter was developed by Brunauer et al. It is evaluated through N_2 adsorption at 77 K, using BET surface area analyzer. The BET (Brunauer–Emmet and Teller) model could be applied to fit nitrogen adsorption isotherm and evaluate the surface area (SBET) of the sorbent [12,1].

4) Ash content

The inorganic content of the carbon was determined using a standard method according to ASTM D 2866 [19]. To determine the Ash content (% ash) approximately 2 g of powdered activated carbon was placed into weighed ceramic crucibles. Carbon and crucibles were dried overnight at 80°C and reweighed to obtain the dry carbon weight. The samples were heated in a muffle furnace 760°C for at 4 h in the presence of air. The crucibles were cooled in a desiccator, and remaining solids (ash) were weighed

[14]. Percent ash was calculated by,

$$\% \text{ Ash} = \left[\frac{\text{remaining solids wt (g)}}{\text{original carbon wt(g)}} \right] * 100$$

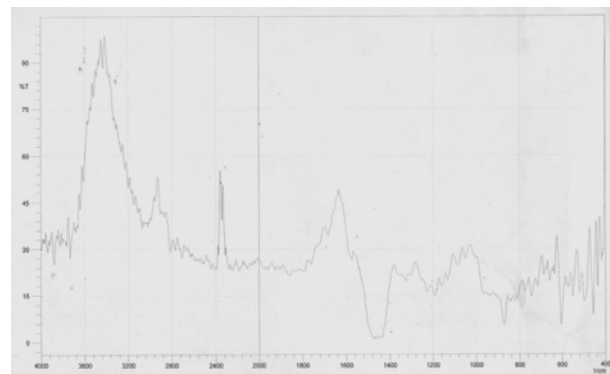
5) FTIR spectroscopy analysis

The carbon surface functional groups are analysed by Fourier transform infrared spectroscopy (FTIR) [23]. Infrared spectroscopy Fourier transform infrared transmission spectra of carbon samples can be acquired using KBr technique, in the wave number range of 4000-400 cm^{-1} . The carbon sample was mixed with KBr at a certain ratio, and the mixture was ground in an agate mortar to very fine powder. After drying in a vacuum oven

at 100°C for one day, the powder was used to make pellet by continuously pressing at 5 ton/cm^2 for 1 min and 17 ton/cm^2 for 10 min with a hydraulic press. The pellet was analyzed immediately after preparation [13].

Fig 4 FTIR spectra of carbon sample

GBC (CT+TT), prepared from 1.5N KOH pretreated gasifier based waste charcoal precursor showed the following absorption bands:



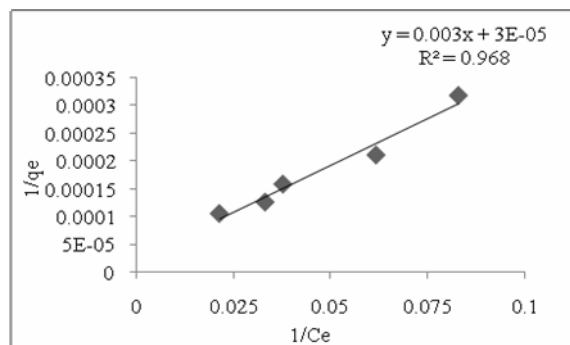
- i) 3486 cm^{-1} : O–H stretching in hydroxyl groups;
- ii) 3450 cm^{-1} : O–H stretching vibrations in hydroxyl functional groups;
- iii) 2972 cm^{-1} : =C–H stretching in unsaturated hydrocarbon;
- iv) 2917 cm^{-1} : –C–H vibration stretching in saturated hydrocarbon;
- v) 2342 cm^{-1} : a sharp characteristic peak of CO_2 while the absorbance arose from its resonance structure of carbon triply bonded oxygen stretching ;
- vi) 1701 cm^{-1} : C=O stretching in ketones and carboxylic acids;
- vii) 1690 cm^{-1} : C=O stretching in ketones and/or in carboxylic acids;
- viii) 1559 cm^{-1} : C=P stretching;
- ix) 1168 cm^{-1} : C=O stretching in ketones and carboxylic acids;

After preliminary study of methylene blue adsorption and due to the problems like, higher settling time, sticky in nature) and cost of the chemical GBC(CT+TT) treated with 1.5N KOH was selected rather than H_3PO_4 treated GBC for the all further study.

4. Results and discussion

4.1 Effect of pH and time

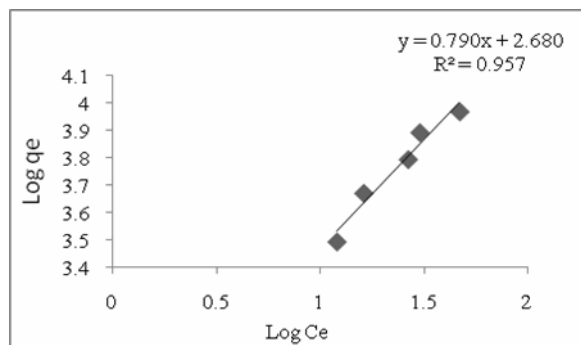
The effect of pH on the adsorption of Pb (II) is presented in Fig. 5. The pH of the aqueous solution is an



important operational parameter in the adsorption process because it affects the solubility of the metal

Fig. 5. Effect of initial pH on the % Removal 50 ppm of Pb(II) metal ions using GBC (CT+TT)

ions, concentration of the counter ions on the functional groups of the adsorbent and the degree of ionization of the



adsorbate during reaction [20]. Thus, the role of hydrogen ion concentration was examined on the Pb(II) removal efficiency. The pH was varied from 2 to 9, other operational parameter initial ion concentration were kept at the optimum and, it is shown that increasing pH of the solution from 2 to 7 caused increase in Pb removal efficiency. The removal efficiency decrease above pH 7. This may be due to the formation of soluble hydroxyl complexes. The hydrolysis of metal ions occurs by the replacement of metal ligands in the inner co-ordination sphere with the hydroxyl group. This replacement occurs after the removal of the outer hydration sphere of metal ions. Adsorption may not be related directly to the hydrolysis of the metal ion, but instead, outer hydration sphere that precede hydrolysis [21].

The equilibration time is one of the parameters for economical wastewater treatment plant applications. Fig. 6 shows the effect of contact time on removal of lead; the adsorption of lead increased as a function of time up to 50 min initial concentration of 50 mg/L and remained almost constant. The rate of sorption of lead on activated carbon was rapid, with 60–70% of lead adsorption occurring within the first 30–40 min and attaining equilibrium was at 70–90 min.

4.2. Sorption isotherm models

The sorption equilibrium data of lead on activated carbon were analysed in terms of Langmuir and Freundlich isotherm models for the purpose of interpolation and limited extrapolation of the data. The Langmuir sorption isotherm equation

Fig. 6. Effect of time on the % Removal of 50 ppm Pb(II) metal ions using GBC (CT+TT); initial pH of solution = 6

$q_e = Q_m b C_e / (1 + b C_e)$ on linearization becomes

$$C_e / q_e = (C_e / Q_m) + (1 / b Q_m)$$

Where, q_e and C_e are the equilibrium concentrations of lead in the adsorbed and liquid phases in $\mu\text{eq/L}$. Q_m and b are Langmuir constants, which are related to sorption capacity and energy of sorption respectively, can be calculated from the intercept and slope of the linear plot, C_e / q_e vs. C_e .

The Freundlich isotherm equation $q_e = k_f C_e^{1/n}$ can be written in the linear form as given below

$$\text{Log } q_e = \text{log } k_f + (1/n) \text{log } C_e$$

where, q_e and C_e are the equilibrium concentrations of lead in the adsorbed and liquid phases in $\mu\text{eq/L}$; k_f and n are the Freundlich constants that are related to the sorption capacity and intensity, respectively. The Freundlich constants k_f and n can be calculated from the slope and intercept of the linear plot, with $\text{log } q_e$ vs. $\text{log } C_e$.

4.3. Effect of initial concentration

The experimental studies were carried out with varying initial metal ion concentrations of lead, ranging from 10 to 50 mg/L using 0.15gm/50ml of adsorbent dose at pH 6.0; the results are shown in Fig. 7 (A) and (B). The results demonstrate that at a fixed adsorbent dose, the amount adsorbed increased with increasing concentration of solution. At lower concentrations, the ratio of number of metal ions to the available sorption sites is low and subsequently the fractional adsorption becomes independent of initial concentration. At higher concentrations, however, the available sites of adsorption become fewer and subsequently the removal of metals depends on the initial concentrations [22].

Fig. 7. Langmuir non-linear (A) and linear (B) adsorption isotherm of Pb(II) at initial pH 6 by using GBC (CT+TT) while initial concentrations were Varied from 50 to 150 mg/L.

Figures 7(A) and 7(B) show the Langmuir and Freundlich (Figure 8) adsorption isotherms of Pb(II), for an initial pH 6, using GBC(CT+TT). The physical parameters for the adsorption of Pb(II) are determined from the linear

Langmuir and Freundlich isotherms and are tabulated in Table 4.

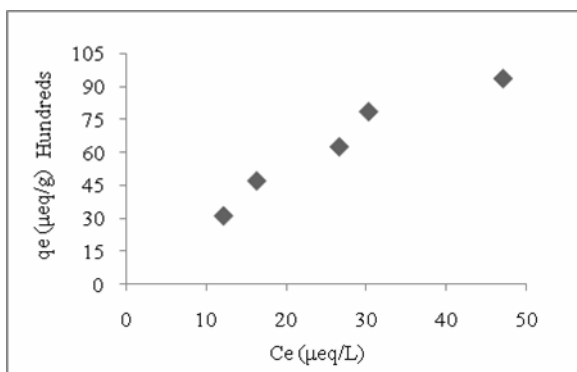
Fig. 8. Freundlich adsorption isotherm of Pb(II) at initial pH 6 by using GBC (CT+TT), while initial concentrations were varied from 50 to 150 mg/L

The values of correlation coefficients (Table 4) of all Langmuir adsorption isotherms indicate that this model fits all the adsorption equilibrium data very well throughout the experimental range of study. GBC(CT+TT) shows higher adsorption capacity as well as stronger affinity towards Pb(II) which is also evident from the isotherms (Figures 7 & 8).

The comparatively higher values of k_f and “n” by GBC(CT+TT) indicate that Pb(II) is more strongly bound onto the surface of GBC(CT+TT). The values of correlation coefficients and model parameters (Fig. 7B and Table 4) of all Langmuir adsorption isotherms indicate that this model fits all the adsorption equilibrium data very well throughout the experimental range of study.

Table 4. Langmuir and Freundlich model parameters for the adsorption of Pb(II) at initial pH 6 and at varying concentrations from 50 to 150 mg/L

Langmuir			
Slope	Intercept	Q_a	b
0.003	0.00003	33333.33	0.01
Freundlich			
Slope	Intercept	k_f	n
0.79	2.68	478.6301	0.79



The adsorption data are also fitted to Freundlich model (Fig. 8 and Table 4), but the better fitting is observed with the Langmuir model.

5. Conclusions

The study indicated that activated carbon prepared from gasifier based solid waste could be used as an effective adsorbent material for the treatment of lead bearing

aqueous wastewater. The adsorption of lead on activated carbon is dependent on contact time, initial concentration, pH. Maximum 96% removal of lead was observed. Removal of about 50–60% occurred in 20-30 min, and equilibrium was attained at around 60-80 min. The values of correlation coefficients and model parameters langmuir adsorption isotherms indicate that this model fits all the adsorption equilibrium data very well throughout the experimental range of study. So, the material (Gasifier based solid waste) under consideration is not only economical, but also a waste product. Hence activated carbon prepared from Gasifier based solid waste would be useful economical treatment of wastewater containing lead metals.

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