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(RESEARCH ARTICLE)

Removal of heavy metals (Cd and Pb) from aqueous solutions by adsorption using synthetic tobermorite prepared from bio-municipal wastes as adsorbent

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Abstract

Contamination of waters by discharge of heavy metals such as Lead (Pb), Cadmium (Cd), Zinc (Zn) etc. from various industrial activities poses a great threat to humans, plants and animals, and thus the need for their removal from waste waters. In this work, synthetic tobermorite was prepared using waste soda-lime-silica glass as silica precursor while snail shell ash was used as source of calcium oxide. Several compositions were formulated with varying combination of soda-lime glass and snail shell ash. The bodies were sintered at 950oC in an electric muffle furnace. Analyses such as scanning electron microscopy (SEM/EDS) and Fourier Transform Infra-red Spectroscopy (FT-IR) was used to assess the microstructure and functional groups present in the developed tobermorite. The synthetic tobermorite was further immersed in aqueous solutions containing heavy metals (Pb2+ and Cd2+) whilst parameters such as temperature, contact time, pH, adsorbent dosage, adsorbate concentration were used to assess the performance of the synthetic tobermorite. The results of the morphology shows that the tobermorites possess irregular and spherical shaped grain with coated water films while the EDS shows the presence of Ca and Si with small amount of Al confirming tobermorite while the FT-IR indicates Ca – O – Si and Si – O – Si as main functional groups. The adsorption experiment performed shows the tobermorite is effective in removal of heavy metals (Pb2+ and Cd2+) studied in this work. However, their performance was impaired at increase in temperature above room temperature.

Keywords: Synthetic tobermorite; Waste glass; Snail shell ash; Heavy metals; Aqueous solutions; Adsorption.

1. Introduction

Over the years, pollution of waters (groundwater, drinking water and wastewater) by heavy metals through discharge from various agricultural and industrial activities has increasingly become an environmental issue all over the world, posing a major threat to humans, plants and animals [1 – 3]. Twenty-three out of about identified thirty-five metals posing a major challenge to human health are known to be heavy metals, among which Pb, Cd, Cu, As, Hg, Ni and Cr are of significant concern as a result of their existence with relatively high concentrations in drinking water and their impact on human health [4, 5]. In view of this, there have been growing interests for removal of heavy metals from contaminated waters [6]. Several remedial methods have been employed over the past few decades in the treatment of heavy metals contaminated aqueous systems. These methods include chemical or electrochemical precipitation, ultrafiltration, ion exchange/adsorption etc. [7, 8]. Despite these developments, there is recently an increasing demand for developments of tailored, low-cost but high efficient non-conventional sorbents from renewable sources, industrial and agricultural wastes for the removal of heavy metals from various aqueous solutions and wastewaters [9].

Tobermorites are a family of naturally occurring hydrous calcium silicate minerals that exhibit selective alkali exchange when replaced with sodium and aluminum [10] and have been known to be an efficient sorbent for removal of divalent

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lead and cadmium ions [11]. However, due to rarity, several methods have been adopted which include sol-gel [12], hydrothermal [13] and sintering reaction [14] using various wastes as precursors. Diatomite and rice husk ash has been used as silica sources [15, 16] while egg shell and marble waste served as precursors for lime [17, 18]. However, little or no detailed works have been investigated on the preparation of tobermorite from snail sell ash and waste soda-lime-silica glass as precursors for calcium oxide and silica respectively and its utilization as sorbent for heavy metals removal from aqueous systems. In this regard, this present study aimed at preparation and characterization of synthetic tobermorite from waste glass and snail shell ash by sintering (solid state reaction) method and its utilization as sorbent for removal of heavy metals (Pb and Cd) from aqueous solutions. Parameters such as pH, temperature, contact time, adsorbent dosage, and adsorbate concentration were used to assess the performance of the synthetic tobermorite while adsorption isotherms were also used to study the mechanism of the adsorption process.

2. Material and methods

2.1. Material and sample preparation

The starting materials utilized in this work are waste soda-lime-silica glass and snail shell ash (SSA) as bio waste. The SLSG is to serve as source of silica (SiO_2) along with other needed oxides while the SS is a precursor for calcium oxide (CaO). The processing of the materials used and their chemical compositions have been reported by the authors' recent work [19]. A total of six (6) compositions (T_bI – TbVI) comprising of varying weight percent mixtures of SLSG and SSA were prepared in this work which are well reported in the authors' recent work [19]. However, only two compositions (T_bII and TbVI) shown in Table 1were utilized for the adsorption experiment. The selection of the two compositions was based on their microstructure, pores and chemical constituents [19].

Table 1 Sample designations by weight percent

	T_bII	**T_bVI**
SLSG	45	65
SSA	55	35

T_b – Tobermorite

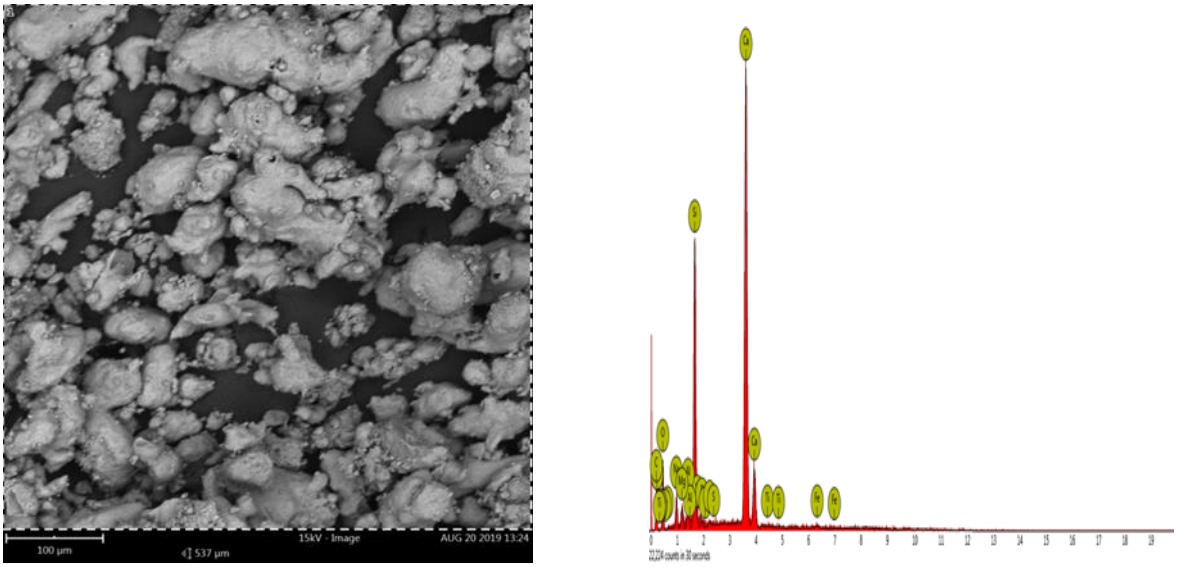
2.2. Batch adsorption experiments

A batch equilibrium experiment was carried out using Pb and Cd solutions in a multi-metal sorption system in order to determine the efficiency of the synthesized tobermorites in removing heavy metals. Cadmium chloride monohydrate ($CdCl_2.H_2O$) of 1.7917g and Lead nitrate ($PbNO_3$) of 1.600 g was dissolved separately in 100 cm^3 distilled water, then later transferred into 1000 cm^3 volumetric flask and made up to mark with double distilled water. 2 g of each studied sorbents (synthesized tobermorites) was equilibrated with 25 ml of prepared multi-metal solutions and agitated at room temperature (25^oC) on a reciprocating shaker to attain equilibrium time which was then used for subsequent experiments. Different solution pH (1, 3, 5, 7 and 9), contact time (1, 2, 6, 12 and 24 h), adsorbate concentration (200, 400, 600, 800 and 1000 mg/L), adsorbent dosage (1.0, 2.0, 4.0, 6.0 and 8.0 g) and temperature (298, 303, 308 and 313K) were used to evaluate the performance of the sorbents (synthesized tobermorites). Two synthesized tobermorites (T_bII and T_bVI) were selected as the used sorbents for the adsorption experiments in this work. Their selection was based on their morphology, their corresponding chemical composition and Ca/Si ratio which might favour better adsorption [19]. Langmuir and Freundlich isotherms were further used to investigate the mechanisms of adsorption process.

3. Results and discussion

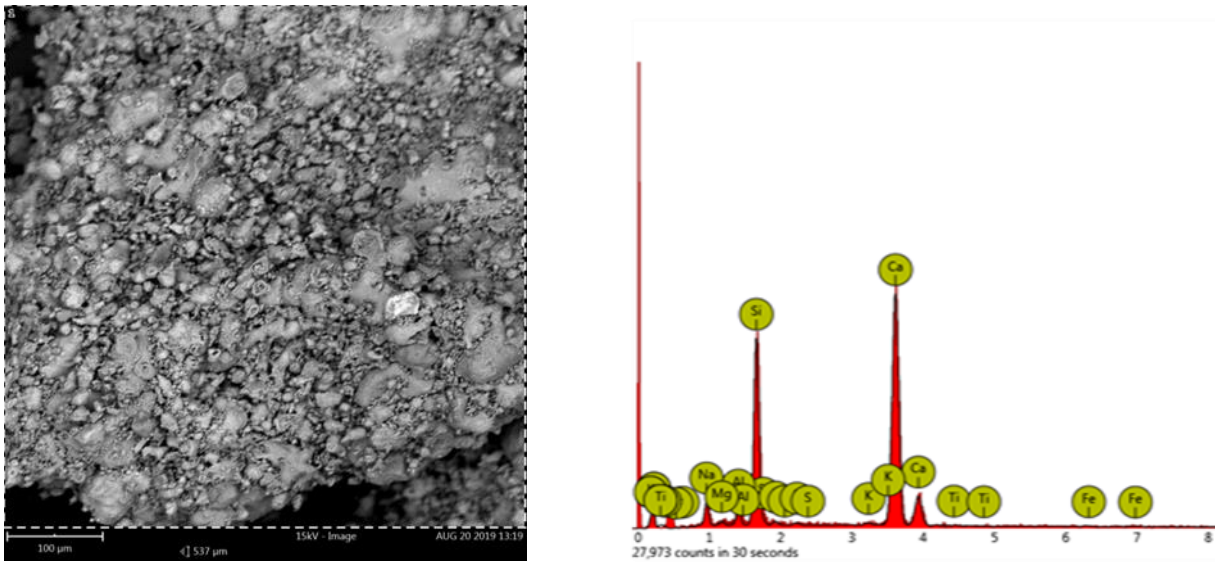
3.1. Morphological characteristics/chemical composition

The results of the microstructure examination investigated by scanning electron microscopy with attached energy dispersive spectroscopy (SEM/EDS) on the synthesized tobermorite samples T_bII and T_bVI are shown respectively in Fig. 1 (a – b). From the SEM micrographs, it can be observed that both synthesized tobermorite particles exhibited similar morphological characteristics as reported by the authors' recent work [19].



Elements	Si	O	Ca	Na	Al	Ti	Fe
(wt. %)	14.09	19.02	48.55	2.25	1.63	0.00	0.00

(a)



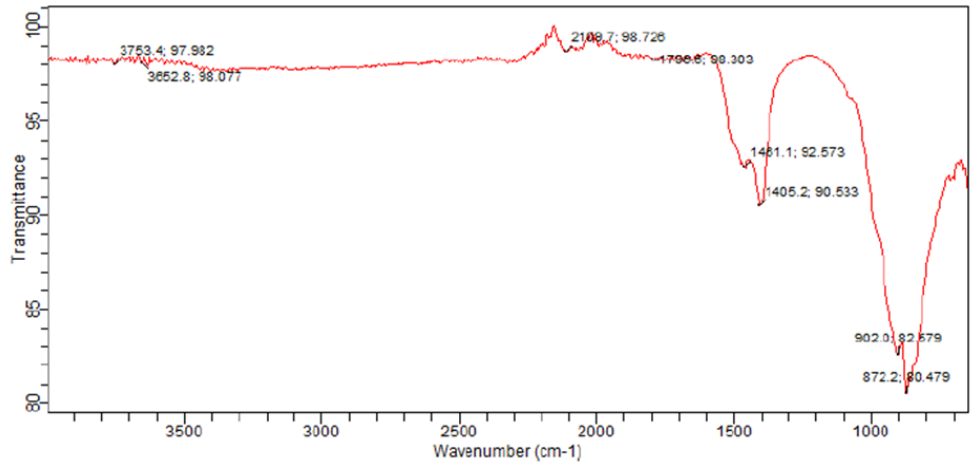
Elements	Si	Ca	Na	Al	Ti	Fe
(wt. %)	24.35	65.79	4.22	2.37	0.00	0.00

(b)

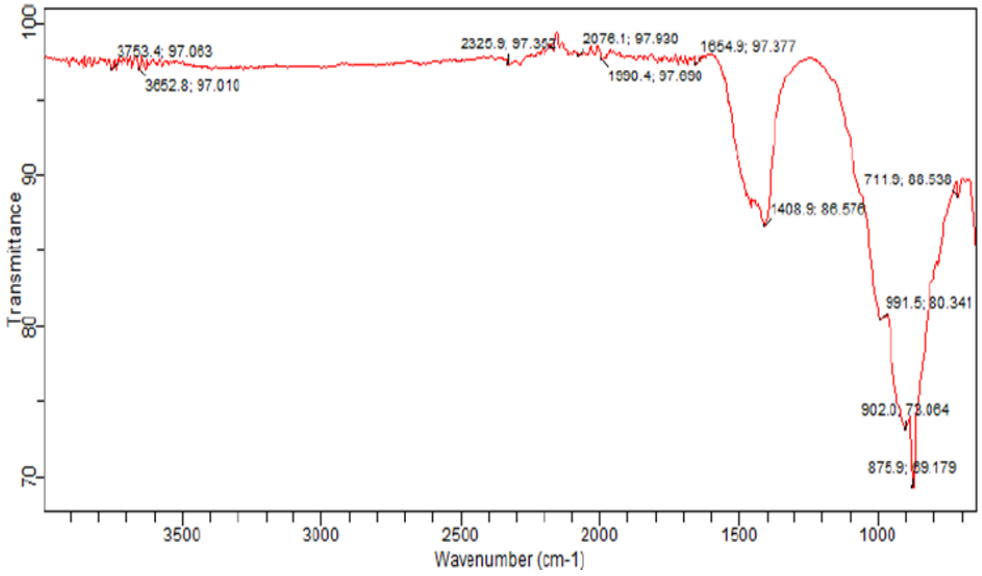
Figure 1 SEM/EDS of synthesized tobermorite samples (a) TbII (b) T_bVI respectively [19].

3.2. Functional group

Figures 2 (a) and (b) shows the diagram of the functional groups present in the synthesized tobermorite samples T_bII and T_bVI using FT-IR as reported by the author [19]. The IR spectra were recorded by FTIR at wavenumber ranging from 500 – 4000 cm^{-1}. The transmission peaks observed between 3753.4 – 3652.8 cm^{-1} might be attributed to the – OH group in calcium oxide as unreacted calcium oxide with water vapor [20]. However, the band in the range 1000 – 850 cm^{-1}can be attributed to Ca – O – Si (calcium silicate) as stated by Meiszterics and Sinko [21]. The intense band 872.2 – 902 cm^{-1} might be due to Si – O – Si. These are reported by the authors in their recent work [19].



(a)



(b)

Figure 2 FT-IR spectra of the synthesized tobermorite for samples (a) T_bII (b) T_bVI [19]

3.3. Influence of parameters on adsorption experiments

Figure 3 represents the influence of pH on the performance of synthesized tobermorite in the adsorption of heavy metals ($Pb^{2+}$and Cd^{2+}). The initial pH of an aqueous solution is an important factor that influences the uptake of the targeted species that are present in the adsorbate. It is observed from Figure 5 that the amount of heavy metals (Pb^{2+} and Cd^{2+}) adsorbed increased with increasing pH up to pH 5 (equilibrium). Similar findings have also been reported in the study of methylene blue adsorption using activated carbon [22]. Ojeme et al. [23] reported that $Pb(OH)_2$ precipitate was formed at pH > 5. At equilibrium pH, two things are bound to happen: extension of the pH range to 8 or 9 could keep the uptake at equilibrium still; or a decline in uptake beyond equilibrium pH could be experienced as a result of further agitation which could force out already adhered ions into the adsorbate since adsorption is a surface phenomenon. Also, a decline in the uptake of Cd^{2+} by the adsorbents at around pH 6 is observed. This significant decline in uptake of Cd^{2+} observed might be attributed to precipitation of Cd $(OH)_2$ at pH ≥ 6 as precipitation has been identified to interfere with adsorption of cadmium ions at that pH according to literature [24, 25]. Ibrahim et al. [26] studied the use of recycled waste glass powder for adsorption of Cd (II), Cu(II) and Pb(II) and found that the maximum metal adsorption was at pH 5.6. As a result, pH 5 can be regarded as the optimum pH for adsorption of heavy metals (Pb^{2+} and Cd^{2+}) using the prepared synthetic tobermorites. Low adsorption of heavy metals at low pH might be due to the presence of H^+ ions competing with the cation groups on the Pb^{2+} and Cd^{2+} for their adsorption site. Also, the electrostatic repulsion between the surface of the synthetic tobermorites and positively charged Pb^{2+} and Cd^{2+} is lowered as a result

of the decrease in surface charged density with an increase in solution pH, thus may result in an increase in the adsorption rate. These further confirm earlier reports that uptake of adsorbate (heavy metals) from aqueous solution by adsorption techniques is depended on the pH of surrounding medium which affect the adsorbent surface charge and the degree of ionization of the adsorbate [27].

Figure 4 represents the influence of contact time on the performance of synthetic tobermorites in the adsorption of heavy metals (Pb^{2+} and Cd^{2+}). It can be observed that the percentage efficiencies for both sample T_bII and T_bVI increase with increasing contact time up to 99.32 % and 99.65 % for Pb removal respectively while 88.55 % and 95.14 % is obtained for Cd^{2+} removal respectively. Meanwhile, Ojeme et al. [23] reported optimum uptake of 78.2% (Pb^{2+}) from aqueous solution using cow dung ash as adsorbent. The rapid adsorption at the initial stage could be attributed to the abundant vacant sites on the surface of the adsorbent [27]. The adsorption equilibrium time occurred around 12 hours, however, T_bVI exhibited better uptake efficiency.

Figure 5 shows the influence of adsorbent dosage as it affects the adsorption of heavy metals. It can be observed that the uptake of adsorbent (percentage efficiency) increased significantly with an increase in the amount of adsorbent (synthetic tobermorite). The increase in the uptake might be due to the increase in a number of active sites owing to an increase in the mass (amount) of the adsorbent [28]. The observed performance is similar in both samples T_bII and T_bVI with maximum uptake achieved at 8 g of each adsorbent used.

Figure 6 indicates the result of the efficiency of the synthetic tobermorites relative to adsorbate concentration (mg/L). It is observed that there is no consistency in the efficiency of the uptake of adsorbate (Pb^{2+} and Cd^{2+}) by the adsorbents (T_bII and T_bVI) as the concentration of the adsorbate increases. The decrease in the adsorption of adsorbate observed as the concentration (mg/L) increases might be attributed to the reduction of vacant sites on the adsorbents as they are completely filled with ions of adsorbate (Pb^{2+} and Cd^{2+}). This indicates that uptake or efficiency of the adsorption process does not depend solely on adsorbent properties, but also on the initial adsorbate concentration (mg/L) in solution [12, 29]. However, T_bVI exhibited better uptake efficiency in Pb removal as the concentration of the adsorbate increases.

Figure 7 represents the influence of temperature on the uptake or efficiency of the adsorbent (synthetic tobermorites). It can be observed that maximum adsorption is obtained at a temperature of 298K for both adsorbents until the efficiency starts to decline steadily at temperature of 303K and above. These phenomena show that adsorption of Pb^{2+} and Cd^{2+} using tobermorite is favored a little above room temperature. The decline in the efficiency as temperature progresses is expected because an increase in temperature (thermal energy) may retard the mobility of the heavy metal ions (Pb^{2+} and Cd^{2+}) in the aqueous solution resulting into desorption [30]. It also indicates an exothermic reaction, which increases the desorption of the adsorbed Pb^{2+} and Cd^{2+} ions [30]. However, both T_bII and T_bVI exhibited excellent uptake performance in removal of Pb until a maximum temperature of 308 K.

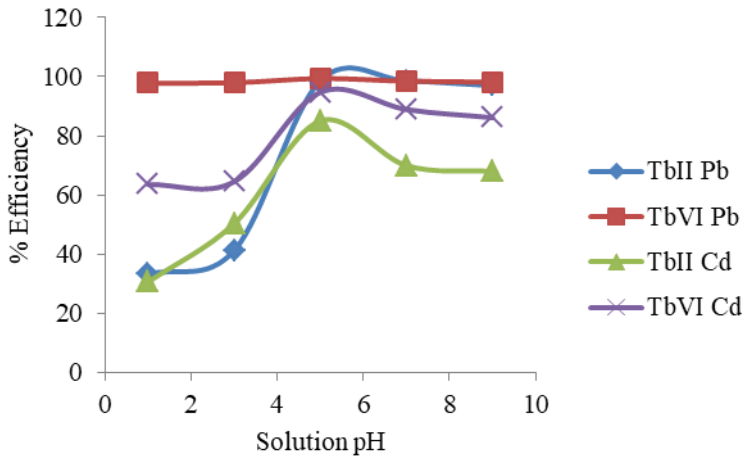


Figure 3 Effect of pH on the heavy metals removal efficiency

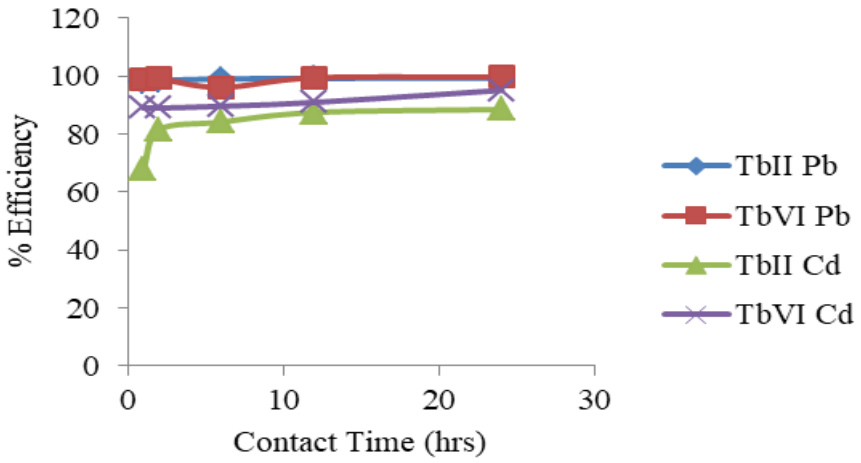


Figure 4 Effect of contact time on the heavy metals removal efficiency

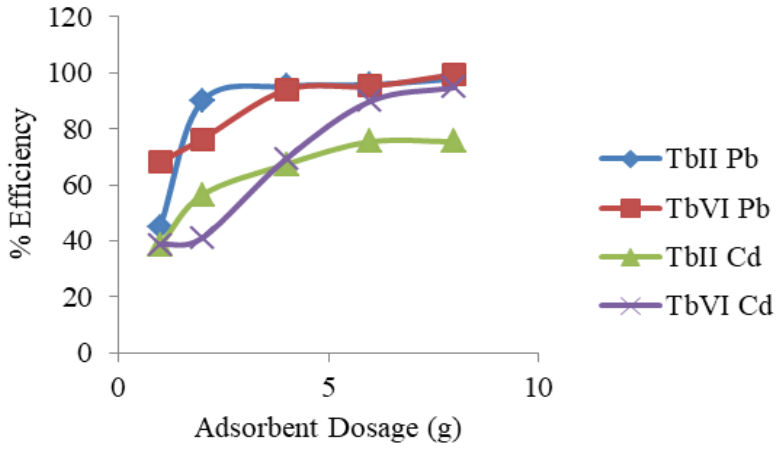


Figure 5 Effect of adsorbent dosage on the heavy metals removal efficiency

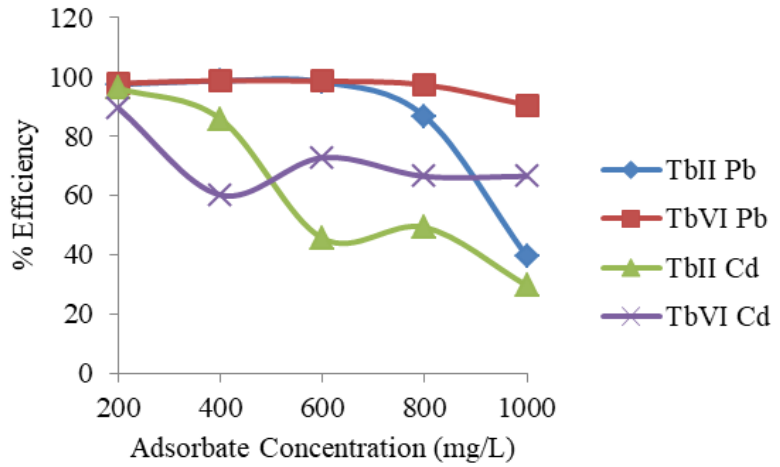


Figure 6 Effect of adsorbate concentration on the heavy metals removal efficiency

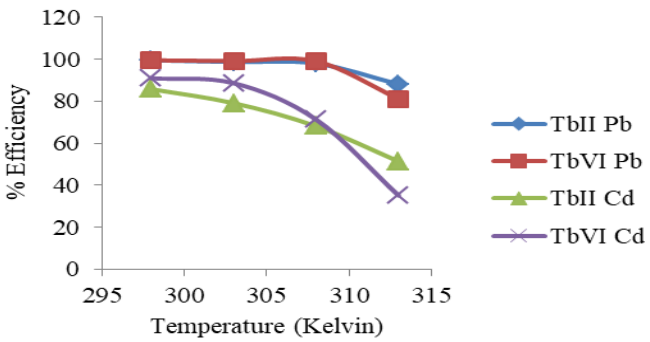
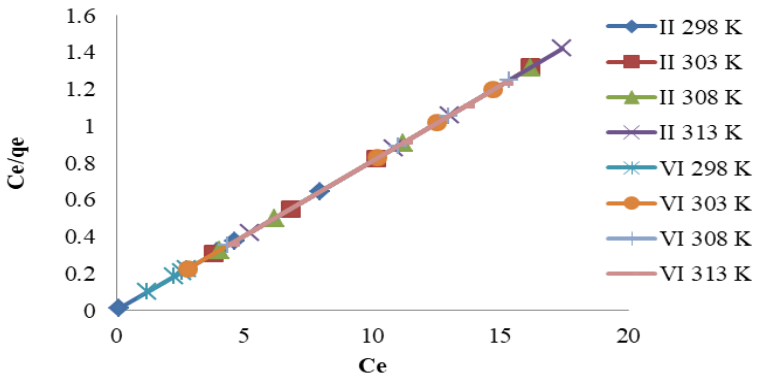


Figure 7 Effect of temperature on the heavy metals removal efficiency

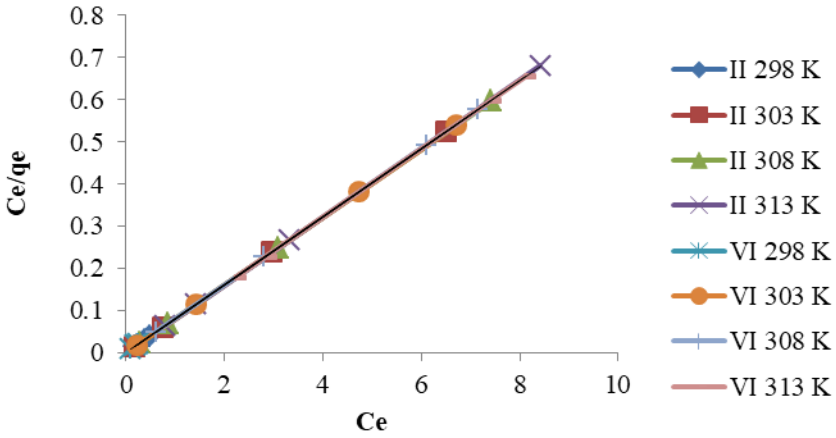
3.4. Adsorption isotherm

The Langmuir adsorption isotherm models for the adsorption of Cd and Pb onto adsorbents T_bII and T_bVI are presented in Figure 8(a) and (b) respectively. The values of q_{max} and K_L (L/mg) obtained from the slope and intercept as well as the R^2 obtained from the linear plots of C_e/q_e against C_e are presented in Table 2. The Langmuir isotherm plots for adsorbents T_bII and T_bVI for the adsorption of Cd^{2+} and Pb^{2+} are linear. From the plots, the correlation coefficient (R^2) of 1.000, which is an indication of a perfect relationship between the adsorption data, was observed for the adsorption of the two metals by the adsorbents at all working temperatures. From Table 2, the values of q_{max} at all temperatures were within the range of 12.270 to 12.500 mg/g which were within the range of experimental value of 12.445 and 12.494 mg/g for the adsorption of the heavy metal by the adsorbents within the working temperature. The values of K_L at decreased with increasing temperature implying a stronger adsorption taking place at lower temperature [31] and also an indication of exothermic processes. Furthermore, the R_L value determines the shape of the isotherm to be unfavorable ($R_L > 1$), linear ($R_L = 1$), favourable ($0 < R_L < 1$), or irreversible ($R_L = 0$). However, the values of R_L at all temperatures are less than 1, indicating that the adsorption favourable at all evaluated temperatures.

The Freundlich adsorption isotherm models for the adsorption of Cd and Pb onto adsorbents T_bII and T_bVI are presented in Figure 9(a) and (b), respectively. The values of q_{max} and K_L (L/mg) obtained from the slope and intercept as well as the R^2 obtained from the linear plots of C_e/q_e against C_e were presented in Table 3. From the plots, the correlation coefficient (R^2) ranges from 0.834 to 0.994 at all the temperatures indicating a strong relationship between adsorption data. From Table 2, the values of n are greater than unity at all temperatures indicated favourable adsorption taking place while the values of $1/n$ tending towards zero confirmed the heterogeneity of the surface. The observed values of K_F, which is an indication of adsorption capacity decreased slightly at a higher temperature, implying an increase in temperature, is not beneficial for the process. Table 3 shows the uptake capacity comparison of the adsorbent developed in this study with other waste-derived sorbents. The comparison however shows that the developed tobermorite in this study showed promising results.

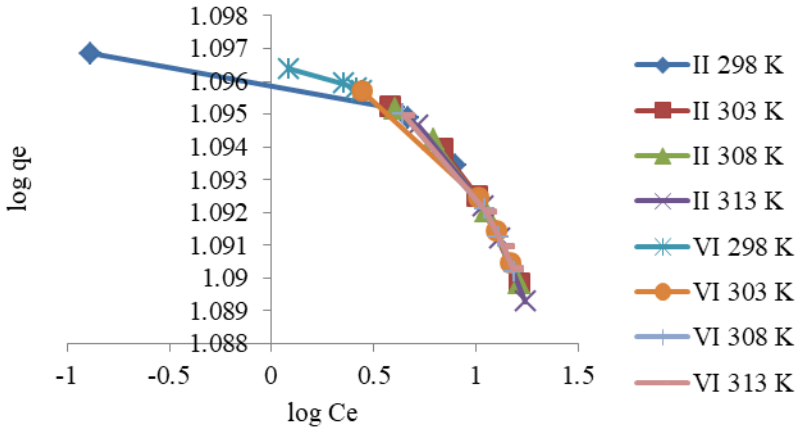


(a)

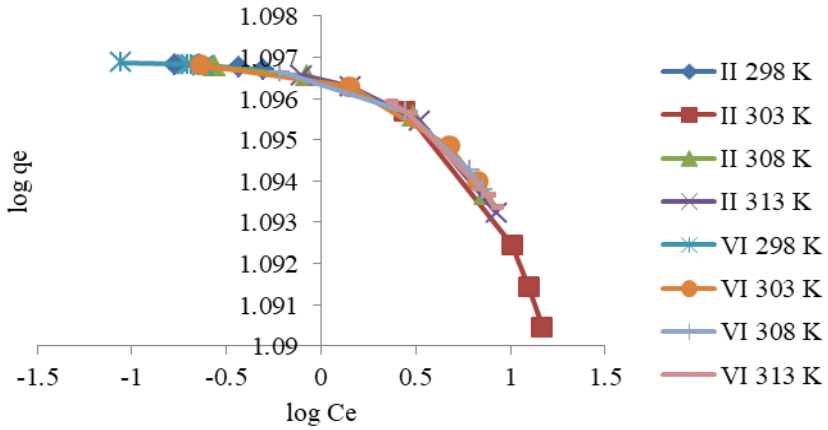


(b)

Figure 8 Langmuir isotherm for the adsorption of (a) Cd^{2+} (b) Pb^{2+}



(a)



(b)

Figure 9 Freundlich isotherm for the adsorption of (a) Cd^{2+} (b) Pb^{2+}

Table 2 Isotherm parameters for the adsorption of Cd^{2+} and Pb^{2+} onto T_bII and T_bVI

Isotherm	**Isotherm Parameter**	**Heavy Metal**	**T_bII**				**T_bVI**			
			298 K	303 K	308 K	313 K	298 K	303 K	308 K	313 K
Langmuir Isotherm	Exp. q_m (mg/g)	Pb	12.498	12.497	12.496	12.490	12.499	12.497	12.492	12.472
		Cd	12.498	12.452	12.450	12.435	12.484	12.465	12.446	12.445
	Cal q_e (mg/g)	Pb	12.484	12.422	12.407	12.376	12.500	12.407	12.407	12.376
		Cd	12.407	12.240	12.255	12.225	12.453	12.285	12.270	12.270
	K_L (L/mg)	Pb	114.295	20.001	16.001	8.031	400.003	20.001	11.4439	5..011
		Cd	114.300	11.986	11.357	9.240	26.700	20.570	13.618	13.854
	R_L ($x10^{-6}$)	Pb	0.008	5.000	6.000	10.000	0.030	4.900	7.000	200.000
		Cd	8.700	80.000	80.000	100.000	3.700	48.000	70.000	70.000
	R^2	Pb	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
		Cd	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
Freundlich Isotherm	1/n $X10^3$	Pb	1/0.3	1/6.8	1/2.1	1/3.2	1/0.1	1/1.8	1/2.6	1/4.6
		Cd	1/1.6	1/8.5	1/8.7	1/9.8	1/1.8	1/6.8	1/8.4	1/8.4
	N $x10^2$	Pb	33.333	1.471	700.280	3.125	100.000	5.556	3.846	2.174
		Cd	6.250	1.177	1.149	1.020	5.556	1.471	1.191	1.191
	K_f	Pb	12.491	12.555	12.474	12.491	12.494	12.474	12.482	12.523
		Cd	12.462	12.607	12.612	12.644	12.488	12.555	12.604	12.604
	R^2	Pb	0.982	0.972	0.850	0.912	0.992	0.862	0.916	0.994
		Cd	0.834	0.952	0.968	0.966	0.994	0.972	0.980	0.984

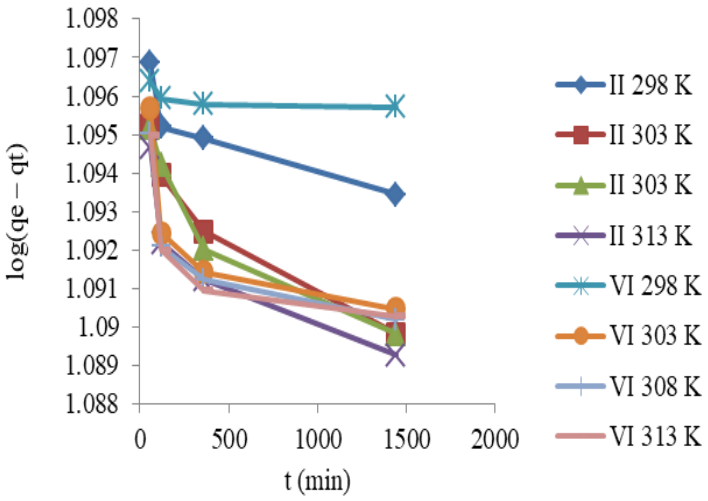
Table 3 Cd^{2+} and Pb^{2+} sorption capacities for a range of waste-derived sorbents

Sorbents	Cd^{2+} uptake (mg/g)	Pb^{2+} uptake (mg/g)	Reference
Synthetic tobermorite	12.44	12.47	This study
Coal fly ash	5.52	2.17	Qi et al. [32]
Recycled waste glass	6.29	11.68	Ibrahim et al. [26]
Waste glass	10.20	10.10	Rashed et al. [33]
Modified chestnut shell	10.14	-	Vazquez et al. [34]
Secondary waste ash	2.92	-	Coleman et al. [35]

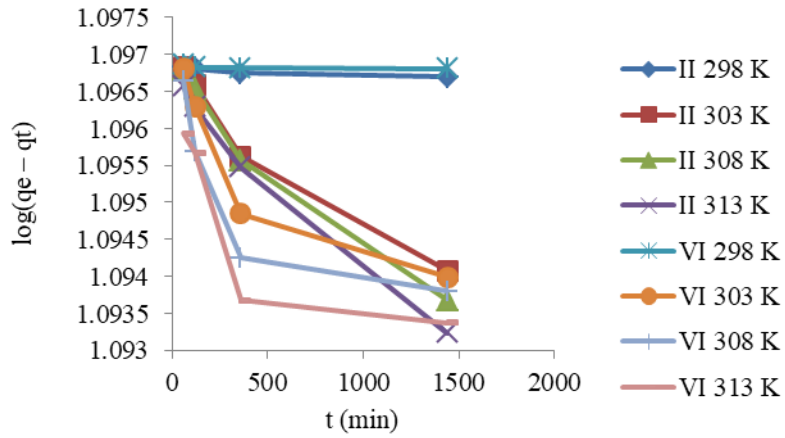
3.5. Kinetic Isotherm

A plot of log ($q_e - q_t$) against t presented in Figure 10 indicates the pseudo-first-order. The values of q_e and k_1 were obtained from the intercept and slope of the plot, respectively and shown in Table 4. From Table 4, the values of R^2 at all temperatures indicating a weak to a strong relationship between data were obtained. From Table 4, the calculated values of q_e are found to be almost of the same values as the experimental values. Also, the values of the pseudo-first-order rate constant, k_1 are observed to be below 1 for the adsorption of the two metals onto both adsorbents at all working temperatures.

The pseudo-second-order expressed by a plot of t/q_t against t is presented in Figure 11. The pseudo–second-order parameters: q_e, k_2 and R^2 were calculated and presented in Table 4. From Figure 11, the R^2 values of 1 are obtained at all temperatures indicating a perfect relationship between adsorption data. Also, from Table 4, the calculated values of q_e have similar values as experimental values. Comparing the values of correlation coefficients obtained for the two kinetic models, it can be said that adsorption processes follow pseudo-second order kinetic model for the concentration of the dye at all temperature. Similar findings have been reported for the adsorption of methylene blue dye on adsorbents.

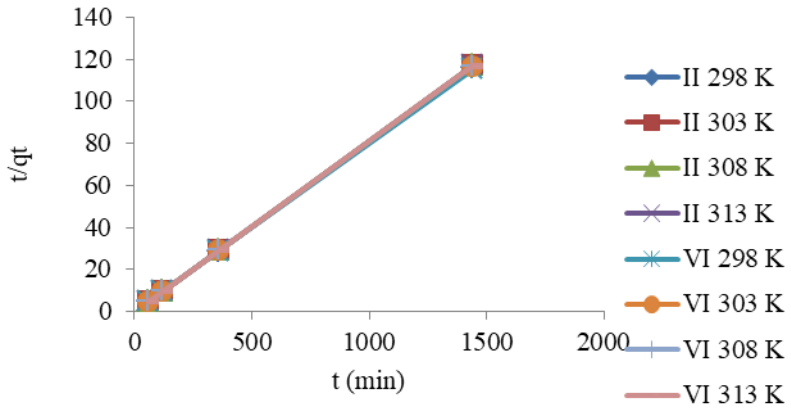


(a)



(b)

Figure 10 Pseudo first order for the adsorption of (a) Cd^{2+} (b) Pb^{2+}



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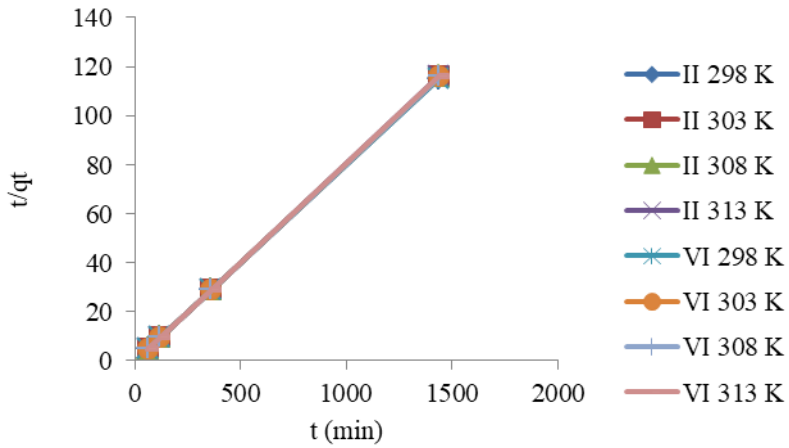

(b)

Figure 11 Pseudo second order for the adsorption of (a) Cd^{2+} (b) Pb^{2+}

Table 4 Kinetic parameters for the adsorption of Cd^{2+} and Pb^{2+} onto T_bII and T_bVI

Kinetic	Kinetic Parameter	Heavy Metal	T_bII				T_bVI			
			298 K	303 K	308 K	313 K	298 K	303 K	308 K	313 K
Pseudo-first-Order of kinetic	q_e (mg/g)	Pb	12.497	12.494	12.494	12.488	12.497	12.457	12.471	12.459
		Cd	12.473	12.434	12.431	12.396	12.477	12.411	12.396	12.459
	K_1 ($x10^{-6}$)	Pb	0.200	4.600	4.600	4.00	0.069	4.600	4.600	4.600
		Cd	4.600	6.900	6.900	6.900	6.900	6.900	4.600	4.600
	R^2	Pb	0.842	0.949	0.968	0.988	0.450	0.777	0.631	0.621
		Cd	0.750	0.902	0.859	0.734	0.433	0.516	0.545	0.481
Pseudo-second-Order of kinetic	q_e (mg/g)	Pb	12.500	12.407	12.407	12.392	12.195	12.405	12.405	12.392
		Cd	12.392	12.285	12.285	12.300	12.469	12.315	12.300	12.303
	K_2 (g/mg/min)	Pb	28.571	1.212	1.029	0.916	117.144	1.578	2.051	2.091
		Cd	1.316	0.669	0.745	0.892	12.531	1.305	1.334	1.591
	R^2	Pb	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
		Cd	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000

3.6. Thermodynamic Studies

The plot of ln *K*C against $1/T$ was prepared and presented in Figure 12. The thermodynamic parameters obtained from the slope and intercept were shown in Table 5. From the Table, negative values of ΔG^0 were observed at all working temperatures for both II and VI adsorbents, indicating that the adsorption processes were spontaneous and feasible. Negative values recorded for ΔH^0 at all temperature confirmed that the adsorption of Pb^{2+} and Cd^{2+} onto II and VI are exothermic. The positive values of ΔS for the two metals onto the two adsorbents showed an increase in the degree of disorderliness at the metals - adsorbents surfaces predicting low energy of attraction between the adsorbents and the metal ions.

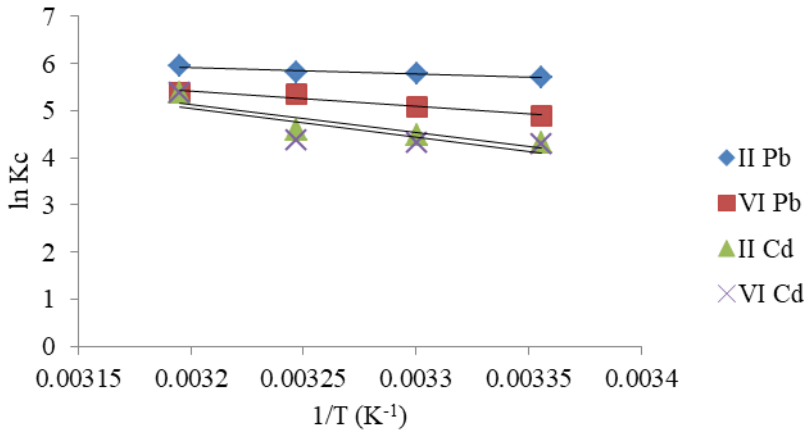

Figure 12 Plot of ln K_c versus $1/T$ for the estimation of the thermodynamic parameters for Cd^{2+} and Pb^{2+}

Table 5 Thermodynamic parameters for the adsorption of Cd^{2+} and Pb^{2+} onto T_bII and T_bVI

Adsorbent	Heavy metal	Temperature	ΔG^0 (KJ/mol)	ΔH^0 (KJ/mol)	ΔS^0 (J/mol/K)
T_bII	Pb	298 K	-15.233	-12.083	10.573
		303 K	-15.286		
		308 K	-15.339		
		313 K	-15.392		
	Cd	298 K	-57.183	-26.339	15.553
		303 K	-57.305		
		308 K	-57.427		
		313 K	-57.549		
T_bVI	Pb	298 K	-30.974	-49.926	24.355
		303 K	-31.051		
		308 K	-31.129		
		313 K	-31.207		
	Cd	298 K	-58.228	-50.888	24.631
		303 K	-58.351		
		308 K	-58.474		
		313 K	-58.597		

4. Conclusion

This research has successfully investigated preparation and characterization synthetic tobermorite ($CaO - Al_2O_3 - SiO_2 - H_2O$) from waste glass and snail shell ash using sintering method and its utilization in heavy metals removal from aqueous solutions. The following conclusions were drawn based on the results obtained:

Bio-waste (snail shell) and industrial waste (soda-lime glass scraps) can be successfully used as precursors for the synthesis of tobermorite instead of using materials that are expensive.

The results obtained from SEM/EDS, FTIR and XRD agreed with one another indicating the presence of calcium silicate hydrate (C-S-H) confirming tobermorite.

The tobermorite is efficient in the removal of Cd^{2+} and Pb^{2+} from aqueous solutions.

The results of isotherm modeling predict that the adsorption is better fit into Langmuir isotherm while the kinetic modeling results indicate that the adsorption follows the pseudo-second order kinetic model.

Thermodynamic studies showed that the adsorption of Pb^{2+} and Cd^{2+} onto T_bII and T_bVI are exothermic, spontaneous and feasible.

Compliance with ethical standards

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Disclosure of conflict of interest

The authors declare no conflict of interest.

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