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Comparing the Efficiencies of the Removal of Cadmium from Industrial Wastewater Using Zeolites

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Authors' contributions

This work was carried out in collaboration among all authors. Author MAA designed the study, performed the statistical analysis and wrote the manuscript. Authors RZ and BSN managed the analysis of the study. All authors read through the final manuscript.

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ABSTRACT

The presence of heavy metals in industrial wastewater needed to be removed in other to control pollution which has been caused as a result of human and industrial activities. Several adsorbents have been used for the remediation process but with this particular research work, Zeolites were used. They were synthesized hydrothermally from various locally available raw materials namely rice husk, corn stalk, corn husk, and kaolin. They were characterized using their FTIR to determine the functional groups on the material synthesized and also their adsorptive activities were compared with industrially synthesized Zeolite X. A very strong peak at 1052 cm^{-1} is as a result of the asymmetric vibrations of the internal T-O tetrahedron with a weak symmetric T-O stretching vibration recorded at 797 cm^{-1}. Another strong peak was observed at 445 cm^{-1} as a result of the O-T-O bending vibration which confirms the presence of a five-membered structure. The adsorptive properties of the synthesized Zeolites were evaluated by using them to remove Cadmium ions from solution and their percentage removals were determined. Similarly, their adsorption kinetics were also determined and was observed the R^2 values to be 0.9093 and 0.9454 for Corn husk and Cornstalk for the first order and the second order with R^2 values of 1 for rice husk, kaolin and corn stalk.

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1. INTRODUCTION

The development of every country is related to the level of Industrial activities it has. This sector provides benefits to humans; it causes long-term environmental problems such as toxicity and pollution discharge their pollutants, for example, toxic gases, nuclear waste [1]. Heavy metals are defined as metallic elements that have a relatively high density compared to water [2] and are also considered as trace elements because of their presence in trace concentrations (ppb range to less than 10 ppm) in various environmental matrices [3]. There are many sources of heavy metals in the ecosystem that come with wastewater streams such as electroplating, smelting, paint pigments, batteries, mining operations and agriculture activities [4]. Factors like industrialization, agricultural activities, urbanization, and population increase are likely reasons for water quality reduction [5,6]. Unlike most organic contaminants, heavy metals are problematic because they accumulate in the tissues of living organisms and do not biodegrade, thereby leading to countless threats to the ecological environments and wellbeing of humans at large [7]. Several adverse health effects have been known for a long time exposure to heavy metals and are even increased recently in some parts of the world, in particular in less developed countries [8]. Among these metals, Cadmium is considered one of the most dangerous heavy metals for human health and the environment [9]. Cadmium (Cd) which is a deadly heavy metal of work-related and environmental worry has been recognized as a substance that is teratogenic and carcinogenic to humans [10]. Cadmium is efficiently retained in the kidney (half-time of 10–30 years) and the concentration is proportional to that in urine [11] and its exposure may occur at lower exposure levels than previously anticipated [12,13]. Several separation techniques such as chemical reduction, electrochemical treatment, ion exchange, precipitation, and adsorption gas bobble separation have been reported for the separation, removal, and control of heavy metals [14,15]. The potential ability of raw fly ash (RFA) and polyelectrolyte-coated fly ash (PEFA) to remove cadmium (Cd) from polluted water has been investigated [10]. The Husk of Melon seed has also been effectively used to remove cadmium from wastewater [16]. Statistical Method was also used for Cadmium (Cd(II)) removal from wastewater by different plant biomasses of rice straw (Oryza sativa) and dragon tree leaves (Dracaena Draco) [17]. The chemical composition of Rice Husk varies depending on factors such as soil chemistry [18,19] climate conditions, paddy variety, use of fertilizer, type of fertilizer [20,21] and year of harvest. Zeolite NaA was synthesized by [22] from Corn Cob ash which was obtained from a company in the United States. In this research, Zeolites were synthesized from raw materials such as Kaolin, Rice Husk, Corn Husk and Corn Cob obtained from the Northern region of Ghana. They were characterized using their FTIR to determine the various functional groups on them and their efficiencies in the removal of Cadmium ions was compared by studying their adsorption studies.

2. MATERIALS AND METHODS

Raw materials such as Rice husk, Cornstalk, Cornhusk, and Kaolin were obtained from the Northern region of Ghana and used for the synthesis of Zeolite. They were pre-treated before used for the synthesis process. Sodium hydroxide pellets were purchased from VWR Chemicals (99%). All the chemicals used were of analytical grade purchased from Sigma Aldrich.

2.1 Preparation of Zeolite from Rice Husk

The pre-treatment process of Rice Husk can be considered in stages involving mechanical, thermal and chemical. Dry raw Rice Husk (RH) was sieved to eliminate residual rice and clay particles [23]. It was washed with water to remove dirt and rinsed with distilled water and dried in an oven at a temperature of 100°C overnight. The metallic impurities were removed with hydrochloric acid [24]. Rice husk was acid leached with 3M HCl for 24 hours, acidified, washed with distilled water and oven-dried at 100°C. It was then calcined in a furnace (Nabertherm at 750°C for 6 hours to eliminate incorporated organic matter to obtain the Rice Husk Ash (RHA). The RHA was mixed with aqueous NaOH and heated at 100°C for 1 hour and filtered to produce Sodium silicate (Na_2SiO_3) solution. Bauxite was pretreated and an appropriate amount weighed and digested in 4M NaOH solution and heated at 150°C for 4 hours in an electrical oven to obtain Sodium aluminate solution. A calculated portion of the Sodium Aluminate from the Bayer Process and Sodium Silicate from the Silica Extraction Process were mixed and stirred vigorously for 1 hour. The resulting gel was homogenized and transferred into a Teflon autoclave and crystallized at 110°C for 24 hours without stirring or agitation for the crystals to precipitate, filtered and dried.

2.2 Preparation of Zeolite from Kaolin

The synthesis of Zeolite from Kaolin started with a pre-treatment process. It was first washed with water to remove soluble organic matter and impurities and then oven-dried at 100°C for 24 hours and calcined at 600°C for 2 hours to obtain the Metakaolin. This was followed by the hydrothermal process as reported by [25,26]. 2 g of Metakaolin was added to aqueous 3M NaOH and was continuously stirred for 1 hour to obtain a homogenous slurry. The mixture was transferred into a Teflon autoclave and placed in an electric oven at 105°C for 2 hours without stirring or agitation for the Zeolite crystals to precipitate. After the crystallization, the Teflon autoclave was allowed to cool to room temperature and the initial pH of the solution was measured. The Zeolite crystals were separated by Vacuum Filtration and were dried overnight at 100°C in an electrical oven.

2.3 Preparation of Zeolite from Corn Stover

The Corn Stalk and Husk materials were cut into smaller pieces, milled and sieved with a mesh size of size 0.0741 mm. It was washed and acid leached with 3M HCl to eliminate metal ion impurities, dried and calcined at 550°C for 2 hours to eliminate incorporated organic matter and other impurities to obtain the pure ash. The acidified ash was mixed with 3M NaOH and refluxed for 2 hours to extract silica in the form of sodium silicate ($NaSiO_3$). This was mixed with sodium aluminate prepared from the Bayer process for 1hour with the aid of a magnetic stirrer to homogenize the gel. The resulting homogenized gel was transferred into a Teflon autoclave and placed in an electrical oven at 110°C for 24hour to precipitate zeolite crystals. The Teflon autoclave was allowed to cool to room temperature and vacuum filtered, washed copiously with distilled water dried for 1hour (due to small quantity) at 100°C in an electrical oven.

2.4 FTIR Spectra of the Synthesized Zeolites

Zeolites produced were characterized by Fourier Transform Infrared (FTIR) to determine the functional groups using the Mattson spectrometer equipped with ZnSe crystal plate and Mercury Cadmium Telluride as the detector and KBr as a beam splitter. The measurements were done using 100 scans at 4cm-1 resolutions, units of log (1/R) which is absorbance over the mid –IR region of 4000-400 cm-1.

2.5 Adsorption Studies of Synthesized Zeolites on Cadmium Removal

2.5.1 Adsorption studies

Synthesized Zeolites were applied to 100ppm and 200ppm concentrations of laboratory prepared Cadmium solution. 1 g of Zeolite powder was added to 100 and 200 ml of the solution and stirred for 4 hours using a magnetic stirrer and samples were taken after time intervals of 5 min, 10 min, 15 min, 20 min, 30 min, 1 hour, 2 hours, 3 hours, 4hours contact time with Zeolite. After filtration, the concentrations of Cadmium in the sample was measured using the Atomic Adsorption Spectrometer (AA 7000 Shimadzu model). This was conditioned by calibrating at 0.05-2.00ppm for five points in 5% HNO_3. The initial calibration value was 1.0ppm. The support gas flow was 15 ml/min and the fuel gas flow was 1.5 ml.min at a wavelength of 228.8 nm and a detection level of 0.0035ppm.

2.5.2 Percentage removal

Percentage Removal of Cadmium by the Synthesized Zeolites was calculated using the formulae:

$$\%R = (Co - Ce)/Co \times 100\% \tag{1}$$

Where;

R% is the percentage recovery of Cadmium from the solution
C_O is the initial concentration of Cadmium in solution
C_e is the concentration of Cadmium in solution at time t.

2.5.2.1 Sorption kinetics

The equations used for the sorption studies were also used by [27]. The Pseudo-first order kinetic model equation is given as:

$$\frac{dq_t}{dt} = k_1 \,(q_e - q_t) \tag{2}$$

By integrating this equation using the boundary conditions were;

t = 0: $q_t = 0$, and t = t, $q_t = q_t$

q_t

$$log\,log\,(\,Cq_e - q_t) = log q_e - \left(\frac{K_1}{2.303}\right)t \quad (3)$$

From (2) $q_e - q_t$qe and q_t are the mass ratios of Cadmium (Cd) adsorbed at equilibrium and at time *t* (mg/g) respectively, t is the contact time (1/min), K_1 is the pseudo-first-order rate constant (/min). This will give a straight-line plot of log (*qe-qt*) against *t* where log (*qe*) becomes the intercept and the slope will be$\frac{K_1}{2.303}$.

$\frac{K_1}{2.303}$ The Pseudo second-order equation is given as;

$$\frac{dq_t}{dt} = k_2\,(q_e - q_t)^2 \quad (4)$$

Using the same boundary conditions as in (2), the integration will give this equation:

$$\left(\frac{t}{q_t}\right) = \frac{1}{K_2\ qe^2} + \frac{1}{q_e}t \quad (5)$$

This will also give a straight-line plot of $\left(\frac{t}{q_t}\right)$ in (mg/g) against t (time.) with $(\frac{1}{q_e})$ as the slope and $\frac{1}{K_2\ qe^2}$ as the intercept and k_2 is the second-order rate constant for the adsorption process.

3. RESULTS AND DISCUSSION

3.1 Fourier Transform Infra-Red Graphs for Synthesized Zeolites

This section describes the various FTIR spectra of the synthesized Zeolites. It shows the peaks at some wavelength (cm-1) which indicate the functional groups on them.

From Fig. 1a. it was realized that the Zeolite prepared from Rice Husk Ash showed clear Zeolitic vibrational bands [28, 29]. The very weak peak at 1630 cm^{-1} is as a result of the bending vibrational mode of Zeolitic water [30]. A very strong peak at 1052 cm^{-1} is as a result of the asymmetric vibrations of the internal T-O tetrahedron with a weak symmetric T-O stretching vibration recorded at 797 cm^{-1}. However, there was a very strong peak at 445 cm^{-1} as a result of the O-T-O bending vibration which confirms the presence of a five-membered structure [31].

In Fig. 1b the FTIR for Zeolite synthesized from Kaolin, the concentration of the NaOH was seen in the asymmetric O-H vibrational bands which were found at 3690 and 3619 cm^{-1} with corresponding very weak symmetric modes at 2020 and 1636 cm^{-1}. The strong asymmetric T-O (where T = Al, Si) vibrational peak of the internal tetrahedral was found at 991 cm^{-1} with a weak Si-O symmetric stretching band at 691 cm^{-1}. Similarly, the ring vibrations and the O-T-O bending vibrations were recorded at 532 and 427 cm^{-1} respectively.

Considering Fig. 1c which is the FTIR for the Cornhusk Zeolite, the short peaks in the region of 1638cm-1 could also be assigned to bending vibration as a result of an adsorbed water molecule [32].

The FTIR graph of the corn stalk Zeolite as shown in Fig. 1d also shows a similar pattern as the Zeolite from Rice Husk and Kaolin. The peak at the frequency of 3343cm^{-1}shows the presence of O-H stretch and 1638cm-1 gives the double bonds of the synthesized Zeolite.

3.2 Percentage Removal

The percentage removal of Cadmium by the various synthesized Zeolites was analyzed and their efficiencies compared.

In Fig. 2 the percentage removal of Cadmium concentration by the Rice Husk Zeolite was very sharp for the 100ppm concentration within the first 30mins. This was followed with a gradual removal for about 20 mins and then a gentle rise in the adsorption process. The percentage removal of the 200ppm Cadmium ions by this Zeolite also showed a sharp increase for about 40mins followed by a decline or desorption within the next 10 mins. This may be due to changes in the pore structure of the adsorbent or the effect of pH .of the solution.

The Zeolite uptake of the 200ppm Cadmium was observed to be very rapid for the first 30-40 mins followed by desorption and then a gradual increase.

The adsorption characteristics of Zeolite synthesized from Kaolin also showed a sharp uptake for the 200 ppm Cadmium solution for the first 30 mins followed by a gentle increase for the next three (3) hours as shown in Fig. 3. This pattern was different from that of the 100ppm where the increase was very steep for the first 5 - 20mins of the process before maintaining a gentle slope.

The percentage removal of Cadmium ions using the synthesized Zeolite from corn stalk as in Fig. 4 was observed to be a better uptake for the 100ppm solution than for the 200ppm which showed a very low uptake just at the beginning and maintaining it throughout the process.

The Cornhusk Zeolite Cadmium uptake in Fig. 5 showed similar initial ionic uptake of Cadmium ions just like the corn stalk zeolite of Fig. 4.

3.3 Kinetics Studies of the Adsorption Process

3.3.1 Graphs for first pseudo-order kinetics

The adsorption kinetics of the various synthesized Zeolites with 100 ppm and 200 ppm Cadmium solution was analyzed to help establish which of the process favors the Pseudo first-order kinetic reaction.

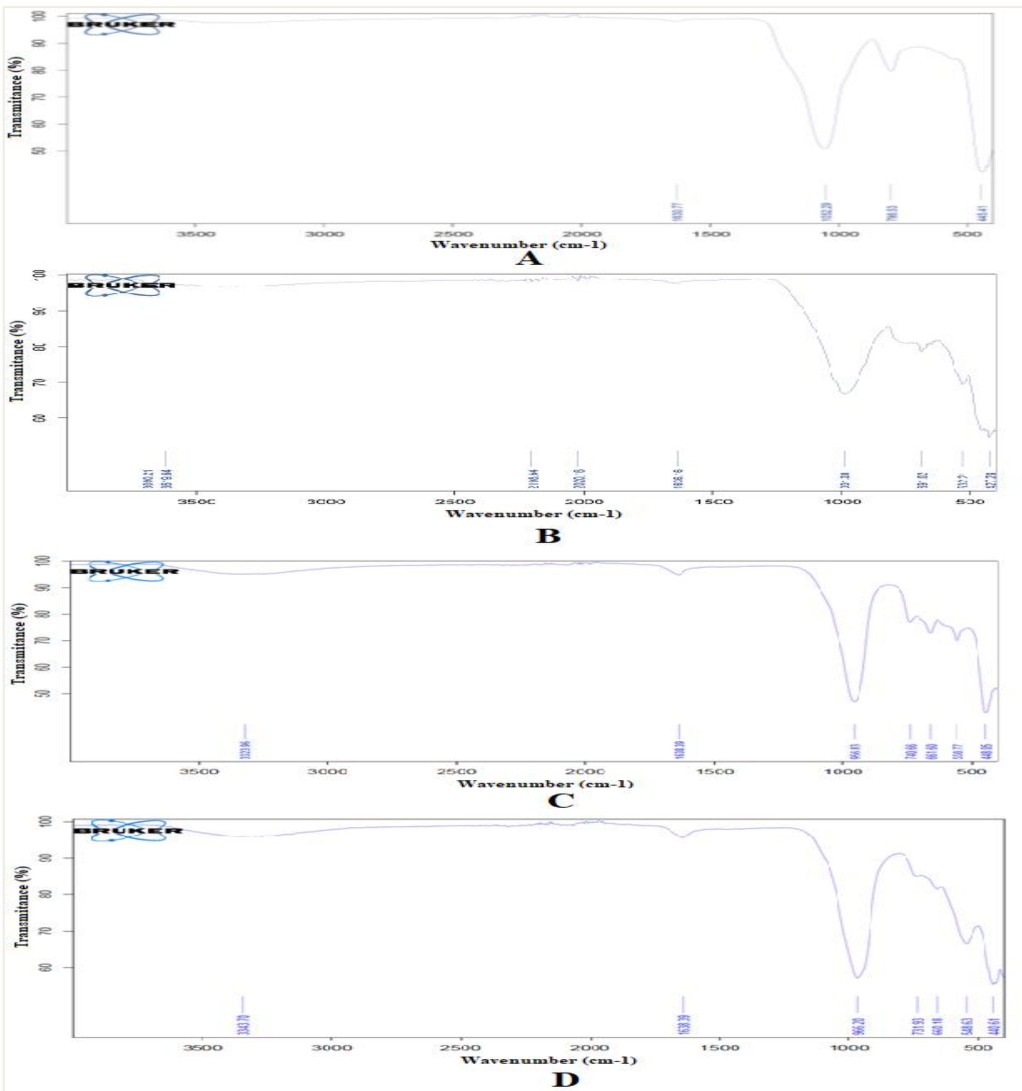

Fig. 1. FTIR diagram of Zeolites synthesized from (a) Rice Husk (b) Kaolin (c) Corn Husk (d) Cornstalk

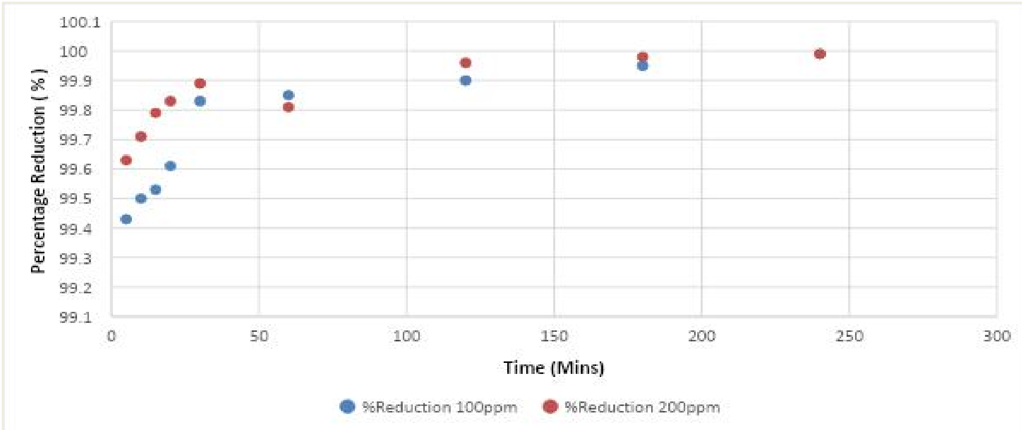

Fig. 2. Graph showing the percentage removal of 100ppm and 200ppm concentration of Cadmium solution using Rice Husk Zeolite

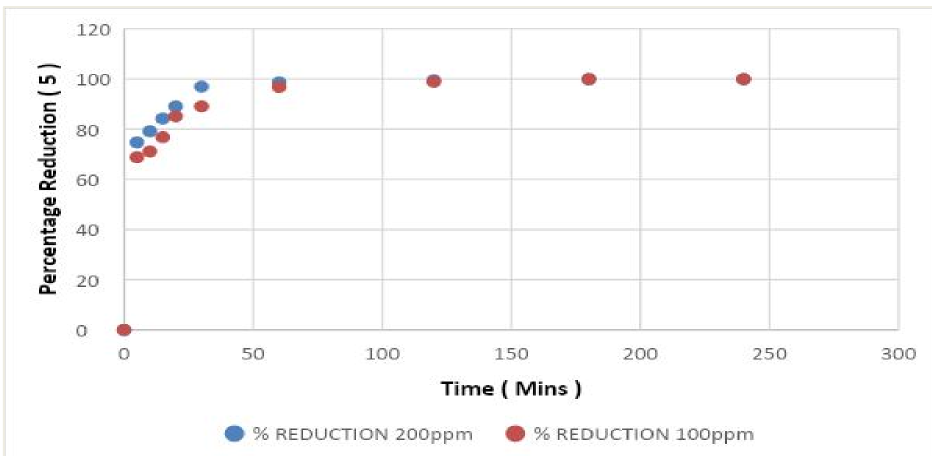

Fig. 3. Graph of the percentage reduction of 100ppm and 200ppm Cadmium solution using Zeolite from Kaolin

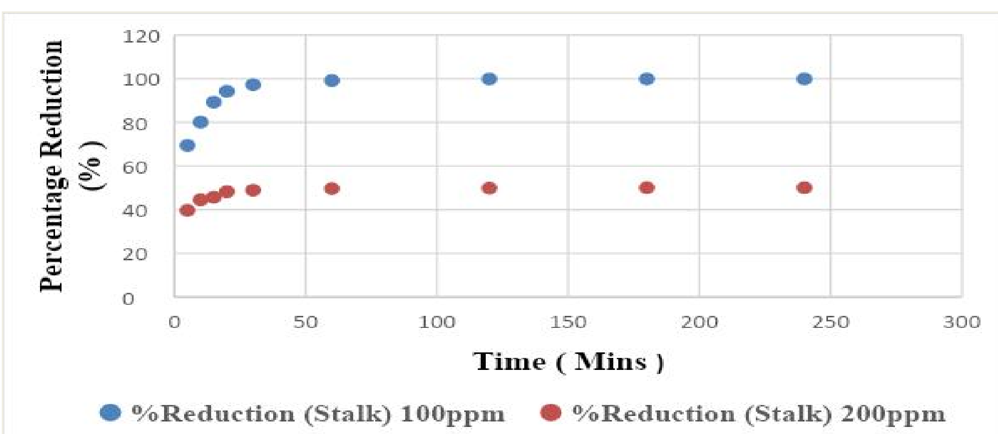

Fig. 4. Graph showing the percentage reduction of 100ppm and 200 ppm Cadmuim solution using Zeolite synthesized from Corn Stalk

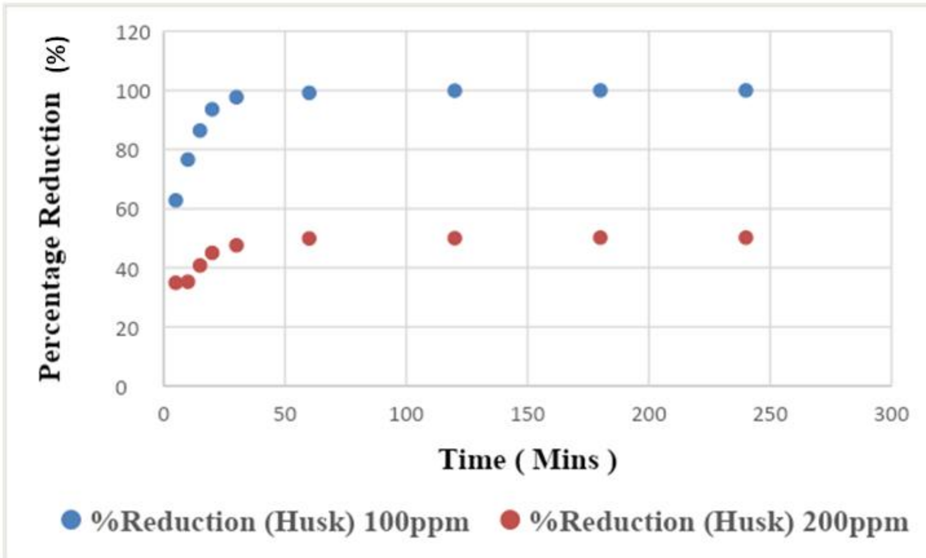

Fig. 5. Graph showing the percentage reduction of 100ppm and 200ppm Cadmium solution using Zeolites synthesized from Corn Husk

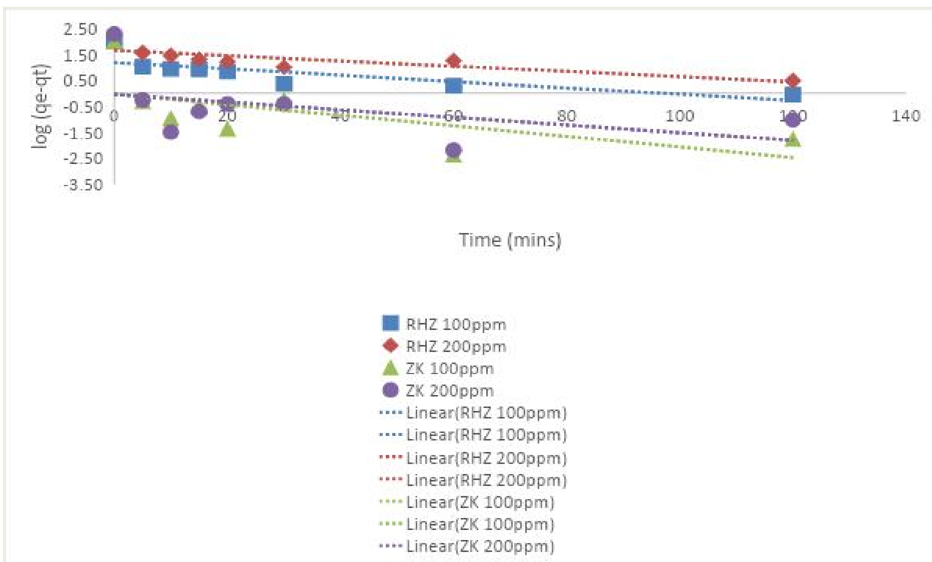

Fig. 6. Pseudo first-order of rice husk and kaolin zeolites

Fig. 6 shows the pseudo-first-order kinetics of the adsorption process for the Rice husk and Kaolin Zeolites for the removal of Cadmium ions in 100ppm and 200ppm solutions. It was realized that the R^2 values were 0.6302, 0.6356, 0.3841 and 0.2014 for Rice husk Zeolite 200ppm, 100ppm and Kaolin Zeolite 100ppm and 200ppm respectively. These values are low and as such their adsorption process does not favor first-order kinetics.

Similarly, Fig. 7 also shows the Pseudo first-order kinetics for Corn husk and Stalk Zeolites. The R^2 values are 0.8361 and 0.7635 for 200ppm reduction using Cornstalk and husk Zeolites respectively. For the 100ppm removal, the R^2 values are 0.9095 and 0.9454 for husk and stalk respectively. This shows that the adsorption process for the removal of Cadmium ion from solution using Zeolite synthesized from Corn husk and Stalk favors the first order.

Fig. 8 also explains the Pseudo first-order kinetics by Zeolite X for the removal of Cadmium ions from the solution. The R^2 value was 0.5113 and 0.5073 for 100ppm and 200ppm respectively and as such does not favor the process.

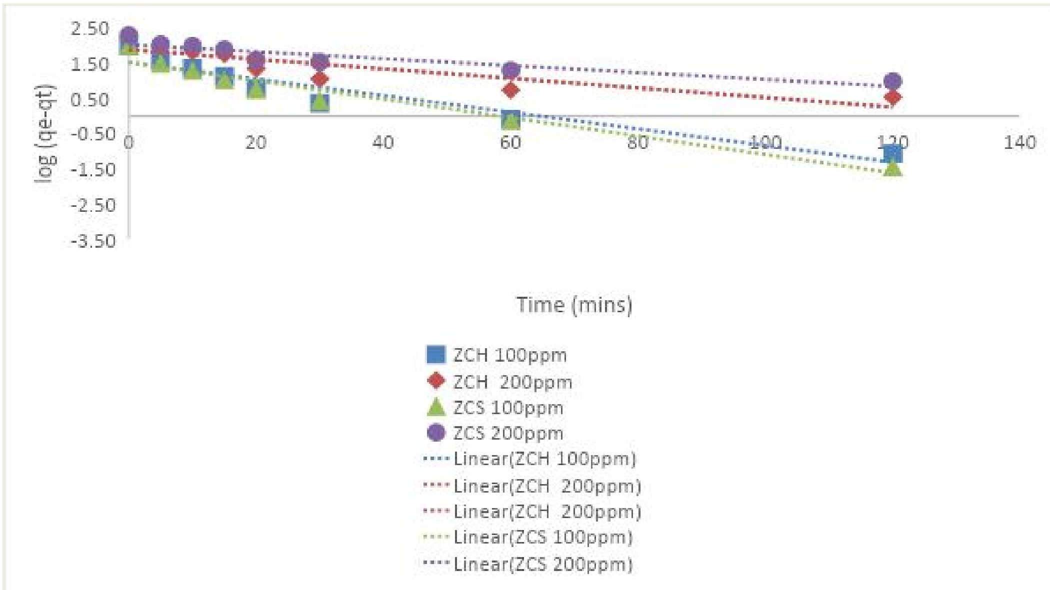

Fig. 7. Pseudo-first order kinetics for corn husk and corn stalk Zeolites

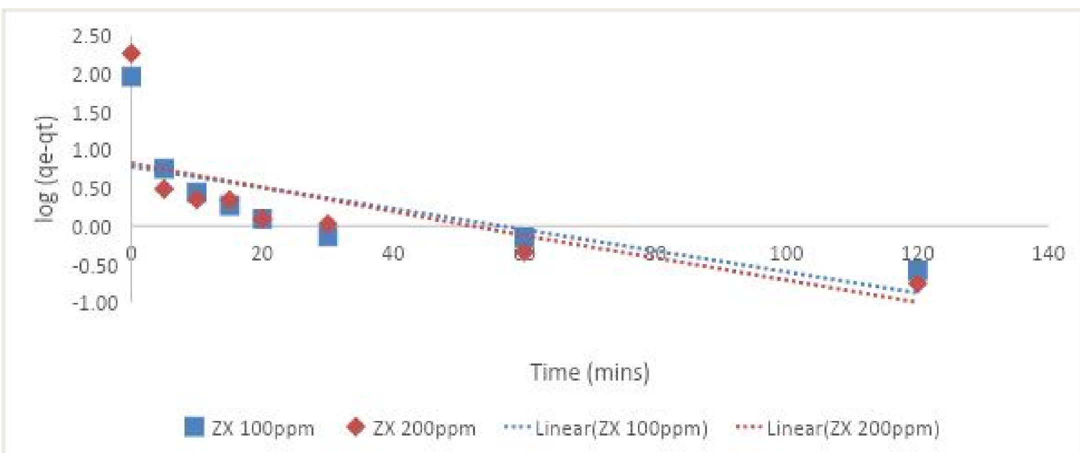

Fig. 8. Pseudo first-order kinetics for Zeolite X

Table 1. A table of the summary for the First-order kinetic model parameters

Sample	100ppm			200ppm		
	R^2	$k_1(min^{-1})$	q_e (mg/g)	R^2	$k_1(min^{-1})$	q_e (mg/g)
Rice Husk Zeolite	0.6356	0.02833	15.7253	0.6302	0.02326	45.9304
Zeolite kaolinite	0.3841	0.04698	0.93907	0.2014	0.03385	0.90469
Corn husk zeolite	0.9095	0.0546	33.8688	0.7636	0.0313	77.2147
Corn stalk zeolite	0.9454	0.06034	33.690	0.8361	0.0228	106.586
Zeolite X	0.5113	0.03178	6.0437	0.5073	0.0364	6.6757

3.3.1.1 First-order kinetic model parameters

Table 1. above shows a summary of the first-order kinetics model parameters for both the 100ppm and 200ppm for the various Zeolites synthesized. Zeolites synthesized from Cornstalk tend to favor the order followed by Corn husk, Rice husk, Zeolite X and kaolin for the 100ppm Cadmium solution. It can also be seen in Table 1. that their R^2 values of 0.9454, 0.9095, 0.6365, 0.5113 and 0.3641 respectively. The highest equilibrium mass ratio (q_e) (mg/g) was 33.8688 mg/g which is for Corn husk Zeolite. A similar trend was also observed with the 200ppm Cadmium solution with Zeolites from Cornstalk having the highest of 0.8361 followed by Cornhusk Zeolite of 0.7636, Rice husk Zeolite of 0.6302, Zeolite X with 0.5073 and kaolin Zeolite of 0.2014. The highest equilibrium mass ratio (q_e) (mg/g) was 106.506 mg/g which is for Cornstalk Zeolite.106.586

3.3.2 Graph for pseudo second order kinetics

The adsorption kinetics of the various synthesized Zeolites with 100ppm and 200ppm concentration Cadmium solution was analyzed to help establish which of the process favors the Pseudo second-order kinetic reaction.

Fig. 9 shows the Pseudo second-order kinetic graph for the adsorption process of Cadmium ions using Zeolites from Rice husk and Kaolin for 100 and 200 ppm concentrations. The adsorption kinetics was very favorable for both concentrations with Rice Husk and Kaolin Zeolite having an R^2 value of 1 and 0.9996 and 1 respectively.

The Pseudo second order kinetics was also analyzed for the Corn and Stalk Zeolites for the removal of Cadmium ions from 100 ppm and 200 ppm solutions as shown in Fig. 10. It was realized that almost all the process favors the order with R^2 values 0.999 and 1 for the 100ppm concentration and 0.999 and 0.9993 for the 200ppm concentrations respectively.

The Pseudo-second-order kinetic adsorption process was also studied for Zeolite X as in Fig. 11 and it was observed that the R^2 values were 0.9996 and 1 for 100ppm and 200ppm respectively.

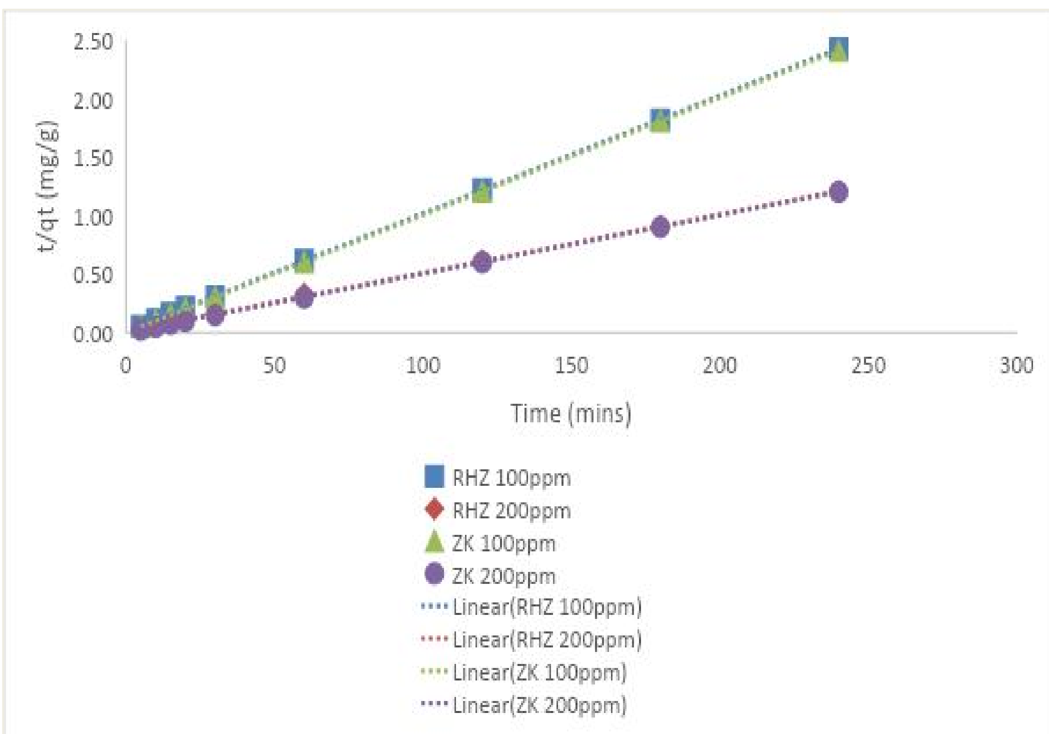

Fig. 9. A graph of the pseudo second order kinetics of Rice Husk and Kaolin Zeolite

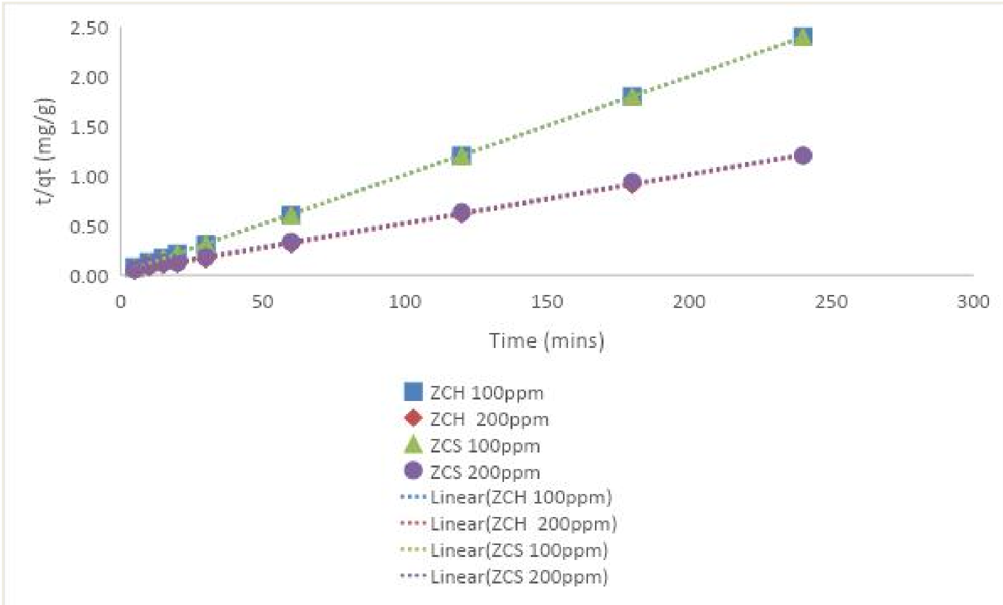

Fig. 10. A graph of Pseudo second order kinetics of Cornhusk and Cornstalk Zeolites

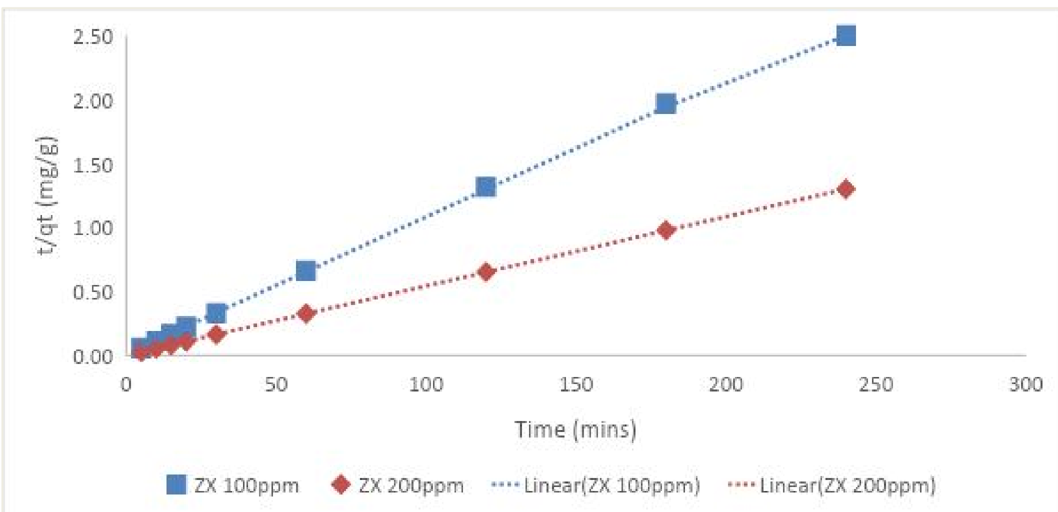

Fig. 11. A Pseudo-second-order kinetics for Zeolite X

3.3.2.1 Pseudo second-order model parameters

Table 2. shows the summary of the second-order kinetics model parameters for both the 100ppm and 200ppm for the various Zeolites synthesized. Zeolites synthesized from Rice husk, Kaolin and Cornstalk tend to favor the order followed by Corn husk and Zeolite X. for the 100ppm Cadmium solution. This can be verified in Table 2. with their R^2 values of 1, 1, 1, 0.999 and 0.9996 respectively. The highest amount adsorbed at equilibrium q_e (mg/g) was 101.0101 mg/g which is for Corn husk Zeolite and Cornstalk Zeolite. A similar trend was also observed with the 200ppm Cadmium solution with Zeolites from Kaolin and Zeolite X having their R^2 values to be1 followed by Zeolites from Rice husk, Cornhusk, and Cornstalk with R^2 values as 0.9996, 0.999and 0.9993 respectively. The highest amount adsorbed at equilibrium for the Pseudo-second-order 200ppm Cadmium solution q_e (mg/g) was 204.0816 mg/g which is for Corn husk and Cornstalk Zeolite.

Table. 2. A table showing the summary of the pseudo second-order model parameter

Sample	100ppm				200ppm			
	R^2	K_2 (g/mg.min)	q_e (mg/g)	H	R^2	K_2 (g/mg.min)	q_e	h
Rice Husk Zeolite	1	0.2	100	2000	0.9996	0.00205	200	81.9672
Zeolite kaolinite	1	0.0083	99.01	81.3008	1	0.25	200	10000
Corn husk zeolite	0.999	0.004712	101.0101	48.077	0.999	0.001154	204.0816	48.0769
Corn stalk zeolite	1	0.0005799	101.0101	59.1716	0.9993	0.000596	204.0816	24.8139
Zeolite X	0.9996	0.009785	93.4579	85.4701	1	0.022431	185.1852	769.23

4. CONCLUSION

The presence of heavy metals in most industrial wastewaters can be reduced through the development of adsorbents using locally available raw materials. This research work showed that Zeolites can be synthesized from sources such as Rice husk, Cornhusk, Cornstalk, and kaolin, and their adsorption capacities compared to an industrially synthesized one (Zeolite X) Zeolites produced were characterized by Fourier Transform Infrared (FTIR) to determine the functional groups and it was realized that they showed Zeolitic peaks. From Fig. 1a a very weak peak at 1630 cm^{-1} which was seen as a result of the bending vibrational mode of Zeolitic water. A strong peak at 1052 cm^{-1} observed is as a result of the asymmetric vibrations of the internal T-O tetrahedron with a weak symmetric T-O stretching vibration recorded at 797 cm^{-1}. However, there was a very strong peak at 445 cm^{-1} as a result of the O-T-O bending vibration which confirms the presence of a five-membered structure In Fig. 1b. the NaOH was seen in the asymmetric O-H vibrational bands which were found at 3690 and 3619 cm^{-1} with corresponding very weak symmetric modes at 2020 and 1636 cm^{-1}. The strong asymmetric T-O (where T = Al, Si) vibrational peak of the internal tetrahedral was found at 991 cm^{-1} with a weak Si-O symmetric stretching band at 691 cm^{-1}. Similarly, the ring vibrations and the O-T-O bending vibrations were recorded at 532 and 427 cm^{-1} respectively. Considering Fig. 1c which is the FTIR for the Cornhusk Zeolite, the short peaks in the region of 1638cm-1 were assigned to the bending vibration as a result of an adsorbed water molecule. The FTIR graph of the corn stalk Zeolite as shown in Fig. 1d also shows a similar pattern as the Zeolite from Rice Husk and Kaolin. The peak at the frequency of 3343 cm^{-1} shows the presence of O-H stretch and 1638 cm-^{1} gives the double bonds of the synthesized Zeolite.

The percentage removal of the Cadmium ion was high for the synthesized Zeolites for the 100ppm solution than the 200ppm as seen in Fig. 2, Fig. 3, Fig. 4 and Fig. 5. Table 1. above gave a summary of the first-order kinetics model parameters for 100ppm for the various Zeolites synthesized shows that Zeolites from Cornstalk tend to favor the order followed by Corn husk, Rice husk, Zeolite X and kaolin. It can also be seen that they have R^2 values of 0.9454, 0.9095, 0.6365, 0.5113 and 0.3641 respectively. The highest amount adsorbed at equilibrium q_e (mg/g) was 33.8688 mg/g which is for Corn husk Zeolite. A similar trend was also observed with the 200ppm Cadmium solution with Zeolites from Cornstalk having the highest of 0.8361 followed by Cornhusk Zeolite of 0.7636, Rice husk Zeolite of 0.6302, Zeolite X with 0.5073 and kaolin Zeolite of 0.2014. The highest amount adsorbed q_e (mg/g) was 106.506 mg/g which is for Cornstalk Zeolite.106.586.

The synthesized Zeolites from Rice husk, Kaolin, Cornhusk, and Cornstalk are comparably effective as the industrially produced Zeolite X.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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