Sorption Kinetics and Diffusion Behaviour of Post-Characterized, Biomodified Waste PET (RIC-1) Bottles Composites

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ABSTRACT

This study dwelt on the preparation, characterization, and application of composites made from waste (PET) polyethylene terephthalate bottles with a resin identification code of 1 (RIC-1). These composites were prepared by melt mixing PET matrix with other fillers such as crab shell, clay, and chitin, resulting in binary or ternary composites. The physicochemical properties and characterization of the composites were analyzed using techniques such as , porosity testing, pH point at zero charge, surface area and FTIR. The composites were thereafter applied for the remediation of iron (III) ions from aqueous solution in which the rate and diffusion dynamics are investigated with the pseudo-first order(PFO), pseudo-second order(PSO) and the Weber- Morris intraparticle diffusion models. The results showed that the pseudo-second order (PSO) kinetic model had a good correlation for all ternary composites, with R^2 values of 0.9977 for PET/CBS/CLAY and 0.9847 for PET/CHITIN/CLAY. The pseudo- first order (PFO) model also showed good correlation, with R^2 values of 0.9812 for PET/CHITIN/CLAY, 0.9276 for PET/CBS and 0.9207 for PET/CHITIN. These results suggest that PSO was more prevalent, and therefore infer that chemisorption was the dominant mechanism for adsorption in these composites. Webber-Morris intraparticle diffusion model applied to data gave a multi-linear correlation of the adsorption of Fe (III) ions on the binary and ternary composites as well as the single materials. However, R^2 values of crab shell, clay, chitin, PET/CLAY, PET/CBS, PET/CHITIN, and PET/CBS/CLAY, were 0.9509, 0.9737, 0.9492, 0.9366, 0.9063, 0.9317, and 0.9169 respectively. The high R^2 values of the rate models shows a positive adsorbent behaviour of the composites.

Keywords: Sorption, kinetics, diffusion, composites, bio-modified,waste PET bottles,

INTRODUCTION

As the global population rises with increasing stiff competition for natural resources there is a projection (Racheeti, 2024) that by the year 2025, half of the world's population will experience a water crisis thus underscoring the need for enhanced water quality and quantity. Numerous scholars, but to mention few, have directed their attention towards the elimination of various noxious pollutants from wastewater (Palani et al., 2021; Abasi et al., 2023; Ali et al., 2019). Heavy metals are known to be one of such pollutants with detrimental impacts on human beings, animals, plants and the environment (Sarma et al.,2024). While certain heavy metals, such as Iron (Fe), Cobalt (Co), Zinc (Zn), and Manganese (Mn), are essential components in metabolism, they can become toxic when present in high concentrations (Fontes et al., 2024; Shafiq and Rehman, 2024;Yu et al., 2024). For example, elevated levels of iron (Fe) found in Epie Creek in Yenagoa city in the Niger Delta made water unsafe for human consumption as well as lowered the clarity and increased turbidity of the aquatic environment (Tarawou et al., 2019). In the Niger Delta region of Nigeria, most groundwater samples (a preferred source of portable water) have a significant amount of iron leading to the abandonment of some already completed boreholes (Ngah and Nwankoala, 2013). (Tariwari et al., 2015) confirmed that the physicochemical parameters of some acclaimed potable water (including heavy metals like iron) was above the Standards Organization of Nigeria (SON) and the World Health Organization (WHO) recommended limit of 0.3 mg/L.The presence of heavy metal toxicity in the environment has prompted the

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exploration of various materials and techniques for the elimination of heavy metals from aqueous solutions. Adsorption has emerged as one of the preferred approaches due to its ability to combine environmental and economic benefits (Chong et al., 2023; Fakhar and Siddiqi, 2023). Both synthetic and natural materials have been utilized in adsorption processes for the remediation of metal pollution. Furthermore, the development of advanced composite materials has received considerable attention in recent years owing to their potential to enhance the properties and performance of various industrial applications (Sunil et al., 2023). Polyethylene terephthalate (PET) is a thermoplastic polymer that is widely utilized, finding its application in the production of soft drink and water bottles and other synthetic products. However, the non-biodegradable nature of these plastics has presented significant environmental challenges. Recycling is one approach to mitigate its impact on the environment; however, it often falls short in addressing the indiscriminate disposal of such plastics. Another approach is to incorporate PET into composites with other materials for various purposes. For example, PET, has been used as a stripping agent for metal ions and other contaminants when blended with fibers, It has also been grafted with different molecules, such as hydrolyzed acrylamide (Rahman et al., 2014), acrylic acid (Niu et al., 2016) and methacrylic acid (Bozkaya et al., 2021) to enhance its adsorption capacity towards metal ions. PET is chosen as the matrix material due to its impressive mechanical properties, however, PET alone may not possess all the desired properties for specific applications, such as porosity for adsorption work. By incorporating materials into the PET matrix, it is possible to improve properties such as porosity which will enhance adsorptive activity of the composite. This study aims at identifying potential adsorptive applications of PET (RIC-1) waste bottles composites on the removal of Fe (III) ions in aqueous solution, after preparation and characterization.

MATERIALS AND METHODS

Reagents and chemicals

The analytical purity of all the substances utilized was guaranteed. Iron (III) nitrate, nonahydrate, Fe $(NO_3)_3 \cdot 9H_2O$ was obtained from Sigma-Aldrich, U.S.A. Molychem of India supplied the sodium hydroxide (NaOH) pellets, which were 97% pure. Hydrochloric acid (HCl) 35-38% (1.18) was obtained from Qualikems Laboratory Reagents, India. Sodium Chloride (NaCl) 99.5% AR/ACS grade was obtained from Loba Chemie PVT. LTD, India.

Preparation of Matrix for Composite Samples

Waste PET(RIC-1) water bottles were collected from drainages, streets, dumpsites and eateries around a University community in Bayelsa state, Nigeria. To get rid of any surface contaminants or commercial labels, the PET bottles were cleaned using a combination of detergent and tap water. The bottles were allowed to air-dry after being rinsed with distilled water. After the bottles were dried and cleaned, they were ground into powder using a stainless-steel grater and then another part was chopped into little pieces using a pair of scissors. The PET powder was then sieved into a fine size by passing it through a 1.0 mm standard sieve.

Preparation of Composite Bio-fillers

Crab shells: The live crabs were gotten from Abalama in Rivers State, Nigeria. The flesh and tissues of the crab were removed from the shell leaving the crab shell clean. The empty shells were washed and sun-dried till there was no more water in it for four days. The sun-dried shells were broken into smaller pieces and taken for grinding and thereafter sieved in a 1.0 mm standard sieve.

Clay: The clay was collected from a depth of 10 - 30 cm with a hand tool from the Abana-enem lake fringe subsurface at Ogu-Atissa, Bayelsa State. It was then oven- dried for two days and sieved in a 1.0 mm standard sieve.

Chitin: Chitin was extracted from crab shells through demineralization and deproteinization. 20 g of the ground and sieved crab shells, hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used in the chitin preparation process. Distilled water was also used to prepare desired concentration of the chemical solutions

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and to wash the sample.

Demineralization experiment: Crab shells contain minerals like calcium carbonate, which needed to be removed to obtain pure chitin. Demineralization was achieved as reported by Aung et al., (2018).This is by treating the ground shells with an acid, such as hydrochloric acid (HCl). The acid dissolves the minerals while leaving the chitin intact. 1M of dilute HCl was prepared and 20 g of the 1.0 mm sieved crab shell was weighed into two separate beakers. The crab shell was soaked in 100 mL of the dilute HCl, stirred continually till frothing subsided. The mixture was allowed to soak for 6 hours. After the 6 hours, the demineralized crab shell was filtered and washed continually till the water ran clean, and then it was oven-dried at a temperature of 105 °C.

Deproteinization: After the demineralization, chitin and protein residues were remaining. To remove the proteins, an alkaline treatment was used. 1M of sodium hydroxide (NaOH) solution was prepared and used to break down and solubilize the proteins, leaving chitin behind. The dried demineralized shell weighing 16.9 g was soaked in 100 mL of the dilute NaOH for 3 hours and heated to a temperature of 105.0(±1) °C. They were washed and filtered till clean. The wet chitin was weighed at a mass of 26.9 g before drying at a temperature of 105.0(±1) and after every 15 minutes the chitin was weighed until a constant weight of 12.1 g was obtained.

Preparation of Composites

A stainless pan was placed on an electric hot plate for 1 minute and 120 g of the shredded PET was weighed and put inside the pot to melt. When the PET was melted at 80 ºC, 30 g of 150 µm of crab shell was weighed and added to the molten PET. The mixture was whisked together to allow uniformity of the blended mixture. This was then poured unto a flat board for curing for about 15 minutes in air. This whole process was repeated for all different composite: PET/CHITIN, PET/CLAY, PET/CLAY/CHITIN and PET/CHITIN/CRABSHELL composites in the ratio 4:1 for the binary composites and 4:1:1 for the ternary composites.

Characterization of Composites

Fourier Transform Infrared Spectrometer (FTIR) Analysis

In order to identify the functional groups that would be employed as adsorption sites for the adsorption of Fe (III)ions, Fourier Transform Spectrometer (FTIR) spectrometer was utilized for all composite analyses.

Physicochemical characterization: Determination of True Density, Bulk Density and Porosity

An empty density bottle was weighed with stopper as W1. The density bottle was filled with solvent (distilled water) and weighed as W2. The density bottle was emptied, and then 2 g of the composite was weighed and transferred into the empty density bottle as W3. Distilled water was added to fill the density bottle containing the 2 g composite and weighed as W4. These measurements were then used to calculate the true density, bulk density and porosity.

Determination of pH point at zero charge (pHpzc)

20 mL of 0.01M NaCl solution was placed in various sample vials and adjusted with 0.1M HCl and 0.1M NaOH solutions, to pHs of 2, 4, 6, 8, 10 and 12 consecutively. 0.2 g of binary and ternary composites were weighed and added to each sample vials and shaken for an hour with a shaker. After 48 hours, the final pH of the solutions was measured. The final pH was subtracted from the initial pH to obtain the change in pH (ΔpH). A plot of ΔpH versus initial pH was drawn to determine the composites.

Determination of surface area

The surface area of the composites was determined using Sear's method (Ebelegi *et al.,* 2023). 0.2 g of each composite sample was weighed into a beaker containing 25 mL of 0.1M HCl. 1 g of NaCl was added and mixed thoroughly. The mixture was titrated with a standard solution of 0.1M NaOH till a pH of about 9 was achieved.

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Batch Adsorption Studies

Effect of Contact Time on Adsorption

0.1g of the adsorbent (single materials, binary and ternary composites*)* were weighed into various test tubes. 60 ppm of Iron (III) nitrate was measured into various test tubes. A speed shaker was used at a constant speed of 250 rpm for various time intervals of 5, 10, 20, 40, 60, 80, 100 and 120 minutes. This was followed by filtration to get the supernatants which were analyzed with a UV Spectrophotometer.

Composite Adsorption, Kinetic and Diffusion Data Analyses

The adsorbed amount of Fe (III) ions on the single materials and composites were calculated using equation (1)

$$q_e = \frac{v}{m}(C_0 - C_e) \quad \text{----------} \quad (1)$$

Where,

q_e = quantity adsorbed, C_o = initial concentration, C_e = final concentration, v = volume of the solution, m = the amount of the single materials/composites

The sorption kinetics was analyzed using the pseudo-first order and pseudo-second order equations (2) and (3) respectively.

$$\ln(q_e - q_t) = -k_1 t + \ln q_e \quad (2)$$

The pseudo-second order(PSO) kinetic model is represented as

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{k_2 q_e^2} \quad (3)$$

Sorption intraparticle diffusion was determined using the Webber-Morris intraparticle diffusion equation (4)

$$q_t = k_{id} t^{1/2} + I \quad (4)$$

The models are plotted with respect to time

RESULTS AND DISCUSSION

The results of the characterization, kinetics and diffusion behaviour of the composites are given in the figures and tables shown in this section.

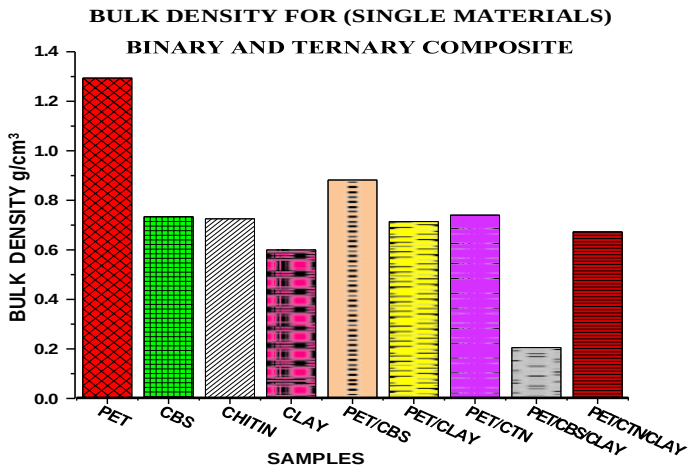

Figure 1: Bar chart for bulk density for the various adsorbents (single materials, binary and ternary composites.)

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Figure 1 shows the result of the bulk density of the (Single materials, binary and ternary composites) considered in this work. PET/CBS/CLAY ternary composite showed the least bulk density value of 0.2054 g/cm^3 while PET had the highest bulk density value of 1.2938 g/cm^3.

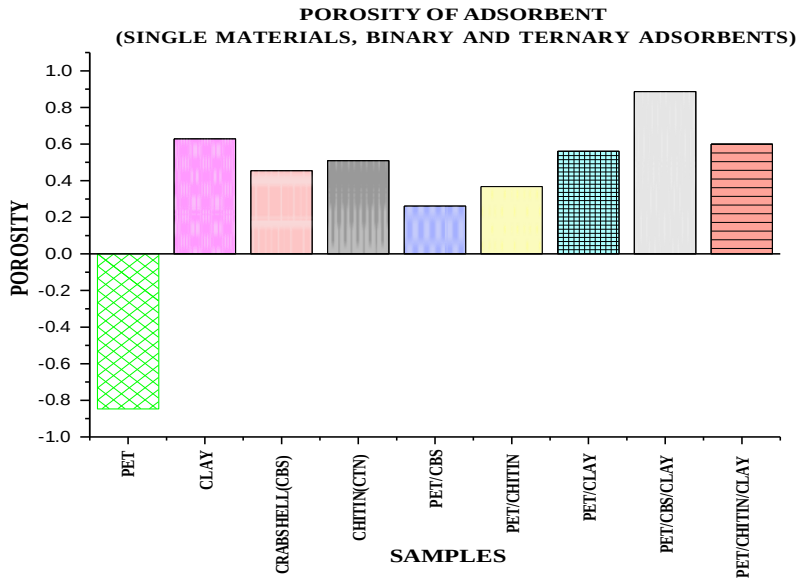

Figure 2: Bar chart of porosity for single materials, binary and ternary composite adsorbents.

Figure 2 depicts the result analysis of porosity test carried out on the various (Single materials, binary and ternary composites) used in this work. The analysis showed that PET had a negative porosity of about -0.0848. Among the single materials, clay showed the highest porosity value of 0.62806 while PET/CBS/CLAY showed the highest porosity value of 0.8868 among all composites considered. The high porosity value of PET/CBS/CLAY observed can be attributed to the low bulk density of the composite material. In contrast, PET low porosity value among the single materials and composites is due to its high bulk density value. The two characteristics are inversely-related.

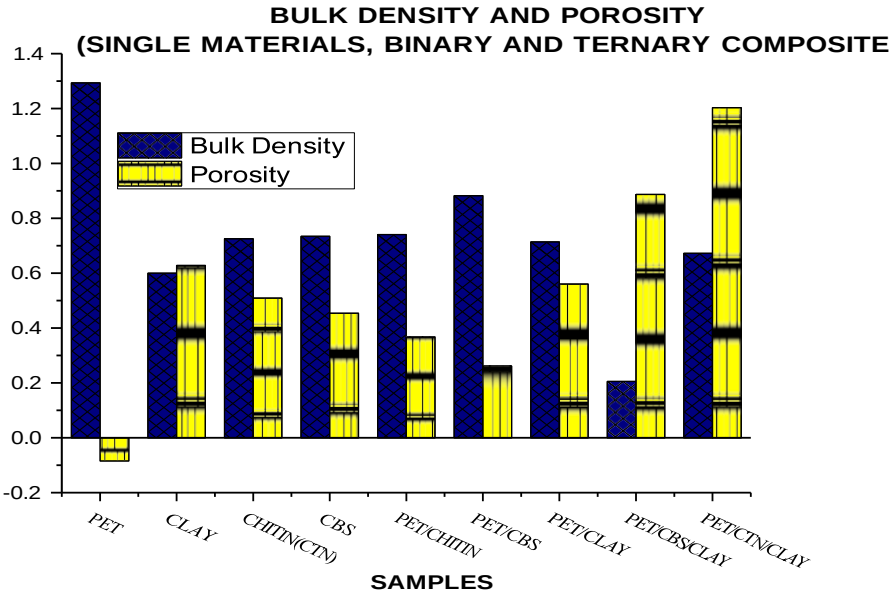

Figure 3: Bar chart of bulk density in relation to porosity

The bar chart in figure 3 shows the relationship between the bulk density and the porosity values. It was observed that single materials or composites with lower bulk densities had higher porosity value as the loosely packed structures created more empty spaces. The binary composites had reduced porosity compared to their single materials. This may be attributed to the introduction of the nonporous and high bulk density PET matrix.

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However, the ternary composites with low bulk densities typically resulted in higher porosity values. The introduction of a second filler material may have contributed to increasing the porosity of the composite.

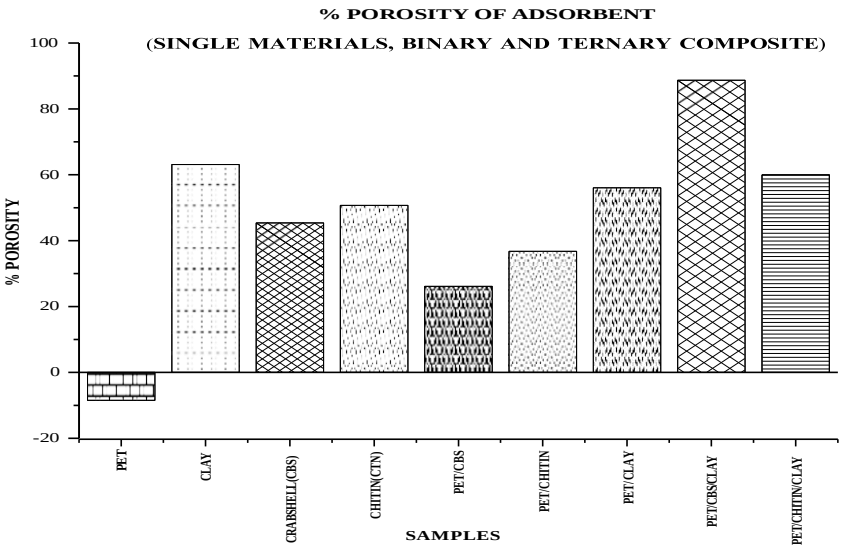

Figure 4: Bar chart of % porosity for the single, binary and ternary composites.

Figure 4 depicts the result analysis of the percentage porosity test gotten from the porosity value carried out on the various adsorbents (Single materials, binary and ternary composites). Here, PET showed percentage porosity values below zero telling us that it possessed little or no porosity. The other single materials as clay, crab shell and chitin showed percentage porosity values of (63.11%, 45.39%, 50.69%) respectively. When the various single materials (Chitin, Clay and Crab shell) where combined either as binary or ternary composite they were seen to improve the porosity of PET. The different composites (PET/CRABSHELL, PET/CHITIN, PET/CLAY, PET/CRABSHELL/CLAY and PET/CHITIN/CLAY) had the percentage porosity of (26.14%, 36.77%, 56.05%, 88.68% and 59.96%) respectively.

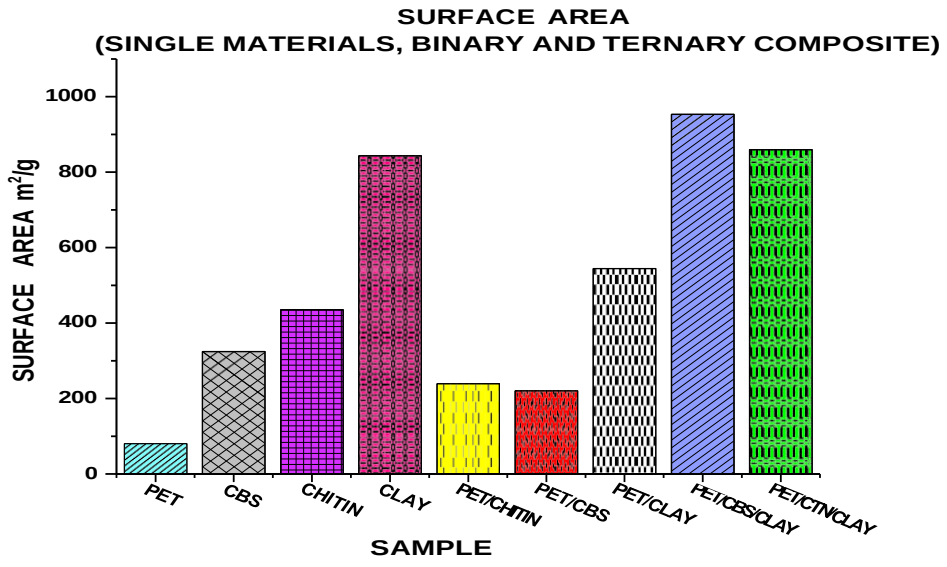

Figure 5: A Bar chart of surface area of the single, binary and ternary composites.

Results from the surface area plot in figure 5 also show PET having the lowest surface area of 80.4 m^2/g which was improved upon when combined as the matrix with other fillers like clay, crab shell and chitin to produce binary and ternary composite yielding values of 239 m^2/g, 220.3 m^2/g, 544.4 m^2/g, 953.13 m^2/g and 859.6 m^2/g for PET/CHITIN, PET/CBS, PET/CLAY, PET/CBS/CLAY and PET/CHITIN/CLAY respectively. The increased surface area among the various composites indicated that there were more available sites for adsorption. It was also observed that the bulk density was inversely proportional to the surface area, as single

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materials like PET with high bulk density values had low surface area while composites like PET/CBS/CLAY with low bulk density value recorded high surface area values.

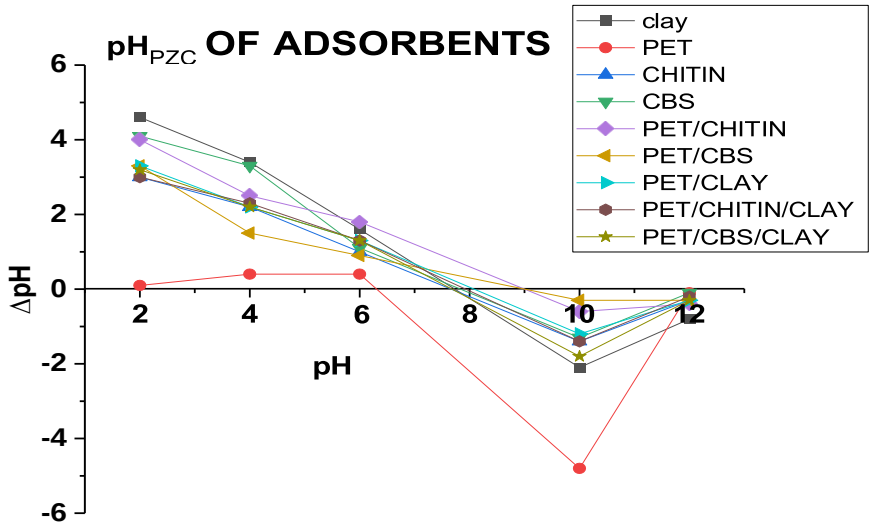

Figure 6: Plot of pH point at zero charge for single materials, binary and ternary composites

Results in figure 6 showed that the single materials had pHpzc values as 6.9, 7.8 and 7.9 for chitin, clay and crab shell respectively.Binary composites had values at 8, 9 and 9 for PET/CLAY, PET/CHITIN and PET/CBS respectively. The two ternary composites both had values at 7.9. Optimized adsorption of metal ions to the composite surface will be achieved at pHs above the observed $pH_{pzc.}$

The Influence of Time on Adsorption

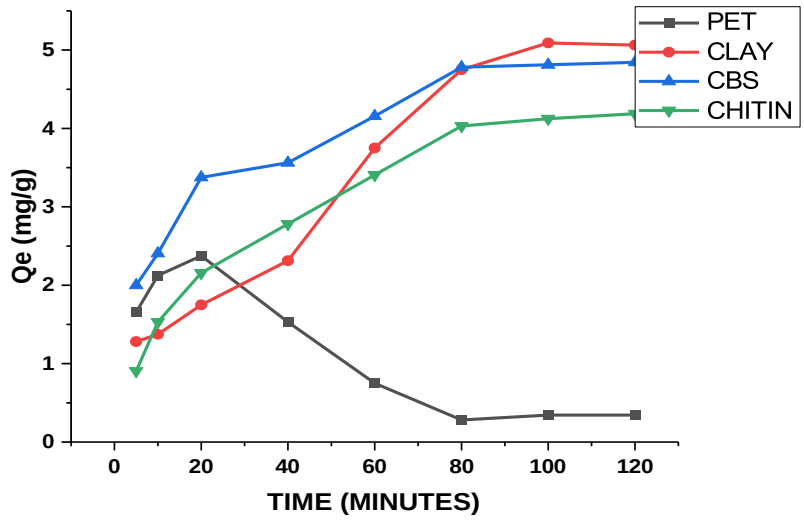

Figure 7: Effect of contact time on the adsorption of Fe (III) using single materials

The adsorption of Fe (III) ions on the various single materials (PET, chitin, clay and crab shell) was investigated varying the time from 5 minutes to 120 minutes. The result from the plot in figure 7 clearly shows a steady rise with time to the point where the curve begins to flatten indicating the time of equilibrium at 80 minutes. However, PET did not follow this trend rather it had fast adsorption from 10-20 minutes and continued to go down supporting the characterization result carried out where PET had a low percentage porosity (-0.0848%) compared to clay, crab shell (CBS) and chitin that had porosity values of 63.11%,

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45.39% and 50.69% respectively. The adsorption capacities of the single materials were all found to be lower than 5.0 mg/g at the time of equilibrium.

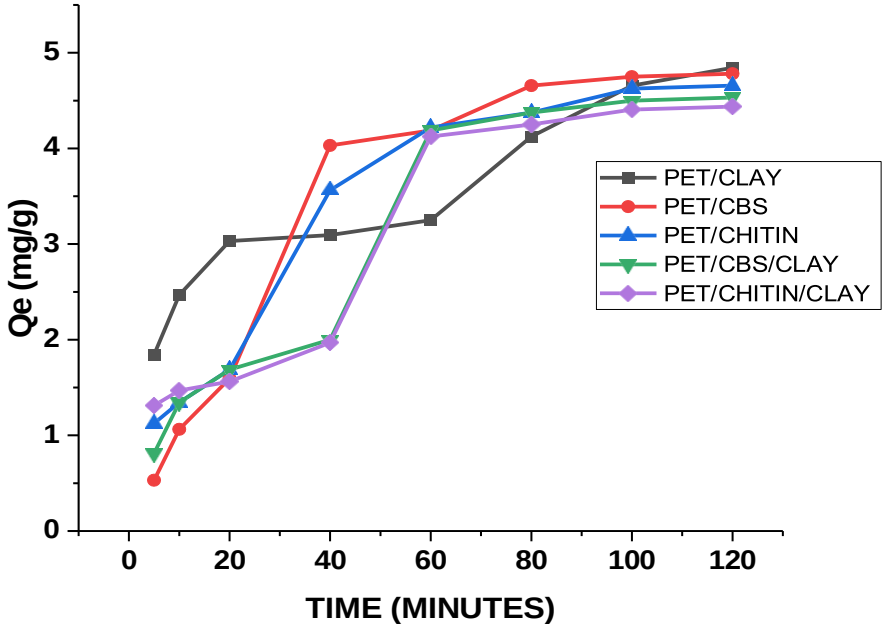

Figure 8: Effect of contact time on the adsorption of Fe (III) using binary and ternary composite.

Figure 8 shows the influence of time on the adsorption of Fe (III) by the binary and ternary composites: PET/CBS, PET/CHITIN, PET/CLAY, PET/CBS/CLAY, and PET/CHITIN/CLAY. The rate of Fe (III) ion removal increased steadily with time until it leveled out, suggesting equilibrium. The saturation of the materials' interior pores and accessible adsorption sites is evidenced by the plateau in the time plot. The adsorption capacities of the materials at equilibrium (q_e) are all lower than 5.0 mg/g.

Kinetics of Adsorption

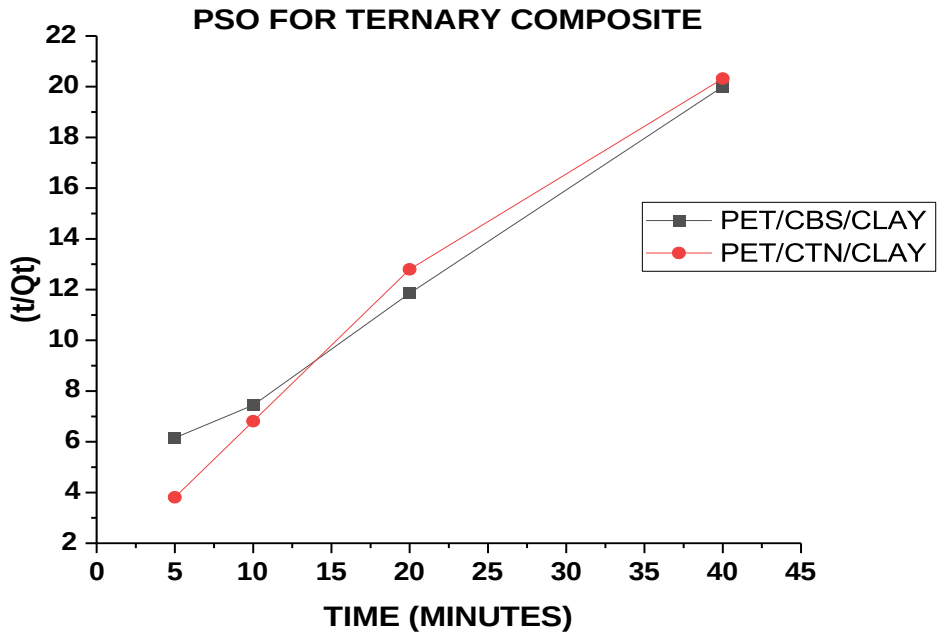

Figure 9: Pseudo-second order plots for ternary composites

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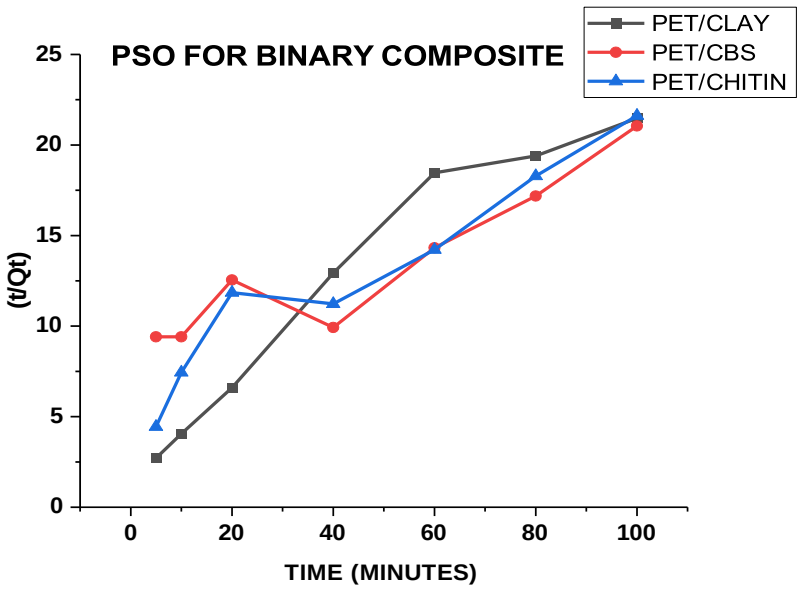

Figure 10: Pseudo-second order plots for binary composites

Table 1: Pseudo-order kinetic parameters for ternary composites

		COMPOSITES	
KINETICS	PARAMETERS	PET/CBS/CLAY	PET/CHITIN/CLAY
PFO	q_e (mg/g)	3.6324	3.1858
	K_1	0.0101	0.0660
	R^2	0.8829	0.9812
PSO	q_e (mg/g)	2.4777	2.1404
	K_2	0.0429	0.1074
	R^2	0.9977	0.9847

Table 2: Pseudo-order kinetic parameters for binary composites

KINETICS	PARAMETER	COMPOSITES		
		PET/CLAY	PET/CBS	PET/CTN
PFO	q_e (mg/g)	2.6469	6.3636	4.6950
	K_1	0.0153	0.0572	0.0348
	R^2	0.7347	0.9276	0.9207
PSO	q_e (mg/g)	3.4519	5.3937	5.4825
	K_2	0.0745	0.0127	0.0092
	R^2	0.9999	0.9966	0.9974

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Intraparticle Diffusion

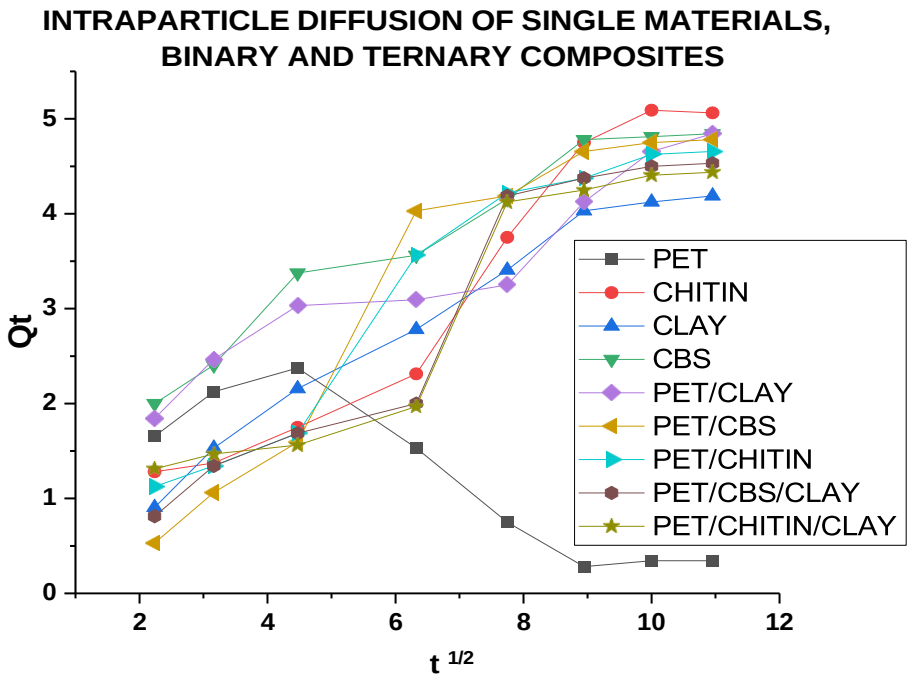

Figure 11: Intraparticle diffusion plot for single material, binary and ternary composites.

Table 3: Intraparticle diffusion parameters for single material, binary and ternary composites.

Sample	**K_{ID}**	**C**	**R^2**
PET	-0.2346	2.7545	0.7899
CBS	0.3354	1.4847	0.9509
CLAY	0.3859	02939	0.9737
CHITIN	0.5066	-0.2377	0.9492
PET/CLAY	0.3156	1.29	0.9366
PET/CBS	0.5352	0.4024	0.9063
PET/CHITIN	0.4633	0.0813	0.9317
PET/CBS/CLAY	0.478	0.2874	0.9169
PET/CHITIN/CLAY	0.4307	0.0431	0.8885

The nature of the adsorption of Fe(III) ions on the binary and ternary composites as well as the single materials determined by the Webber-Morris intraparticle diffusion in the graph in figure 11 showed multi-linearity. This observation according to Olu -Owolabi et al., (2014), shows that the Webber-Morris intraparticle diffusion was not the rate controlling process of diffusion. Nonetheless, the intraparticle diffusion for crab shell(CBS), clay, chitin, PET/CLAY, PET/CBS, PET/CHITIN, and PET/CBS/CLAY, respectively, had a high R^2 value of 0.9509, 0.9737, 0.9492, 0.9366, 0.9063, 0.9317, and 0.9169, which was almost equal to unity. This indicates that the intraparticle diffusion was significant in the adsorption process of Fe (III), but not the controlling step of the adsorption.

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FTIR Spectroscopy Characterization of Composite

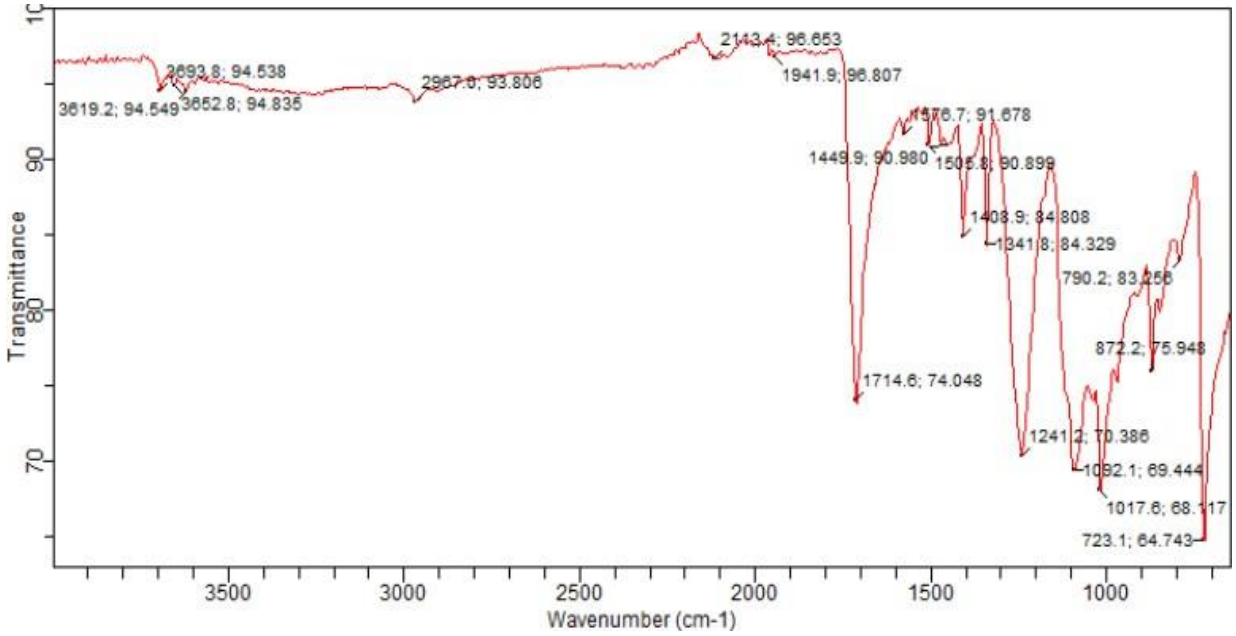

Figure 12: FTIR of PET/CBS/CLAY (Pre-Adsorption)

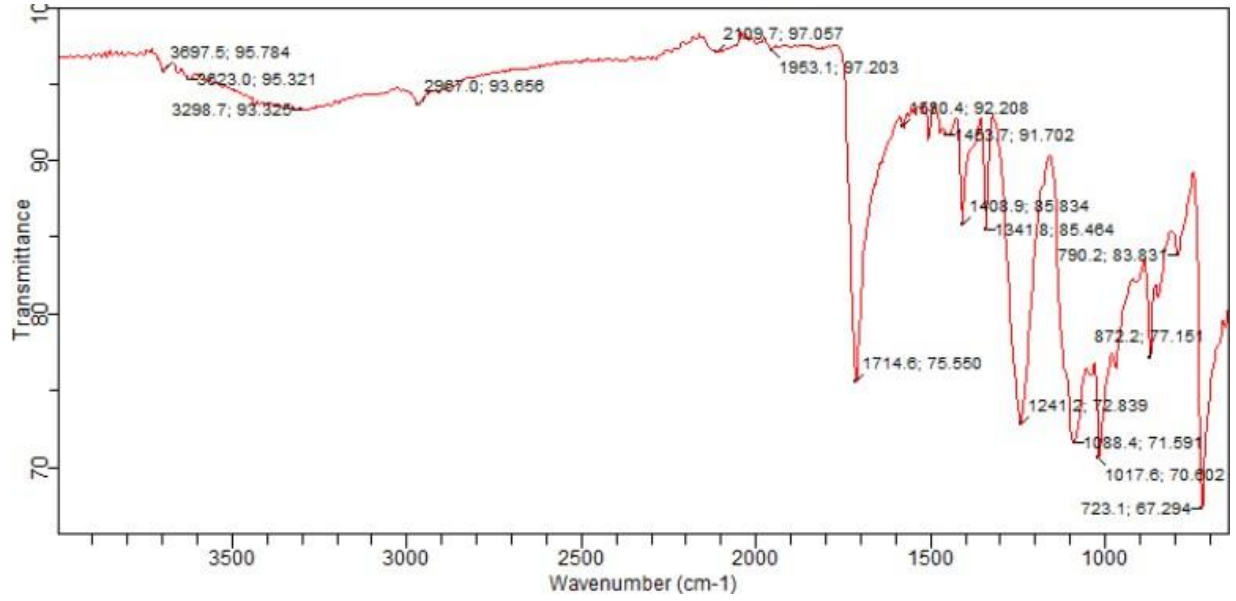

Figure 13: FTIR of PET/CBS/CLAY (Post-Adsorption)

Table 4: Fourier Transform Infrared Spectroscopy Characterization

SAMPLES	**Adsorption Bands (cm^{-1})**		**Inference**
	Pre-adsorption	**Post-adsorption**	
PET/CBS/CLAY	3652 2967 1714 1241-10821	3623/ 3298.7 2967.0 1714.6 591 and 602	Both samples of composites indicated OH, C-O, C=O functional groups.

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The FTIR spectroscopy is a spectral chart that shows peaks and bands consistent with materials containing alkanes, alkenes, alkanols, carbonyls and aromatic functional groups (Ghadah and Foziah, 2018). The result of FTIR analysis for PET/CBS/CLAY sample showed that at wavelength of 3625cm^{-1} for pre-adsorption sample, free OH was noted. Post-adsorption sample of PET/CBS/CLAY also showed more clusters of OH at wavelengths of 3623cm^{-1} (free OH), 3298cm^{-1} (OH bonded), 2967cm^{-1} (OH bonded). PET/CBS/CLAY also showed C-O functional group at a wavelength of 1241.2cm^{-1} with a transmittance of 66.977% in contrast to 88.813% transmittance observed for the post-adsorption sample at the same wavelength. The functional groups present in the composite were sites for the adsorption of Fe (III). Adsorption of ions changed the chemical structure of the materials, which changed their interaction with infrared light, as shown by the observed changes in transmittance values and wavelengths between the pre- and post-adsorption samples of the composite. The findings of the FTIR analysis for the post-adsorption samples showed adsorption peaks at 591cm^{-1} and 602cm^{-1} for PET/CBS/CLAY all indicative of stretching vibrational mode of Fe-O bonds.

CONCLUSION

The preparation, characterization, and application of composites made from waste (PET) polyethylene terephthalate bottles with a resin identification code of 1 (RIC-1) and clay, crab shell and chitin were carried out. These composites prepared by melt mixing PET matrix with other fillers such as crab shell, clay, and chitin, resulted in binary or ternary composites. The porosity of PET (RIC-1) waste bottles, which are recognized as environmental pollutants which are impervious, was increased in this work by using clay and crab shells and chitin. The enhanced porosity of PET (RIC-1) when combined with fillers like crab shell, clay and chitin made it a viable adsorbent material for adsorption of Fe (III) in aqueous solution. The ternary composite PET/CBS/CLAY had the highest porosity result (0.8868 or 88.68%) after characterization. The pH_{pzc} result of the most adsorbing composite PET/CBS/CLAY showed that optimal removal of Fe(III) can be obtained at buffered pHs slightly above the pH_{pzc} (7.9). The adsorption of the single materials and composites varying the time from 5 to 120 minutes, showed a steady rise with time until it began to flatten at an equilibrium time of 80 minutes. The adsorption capacities of the materials at equilibrium (q_e) were lower than 5.0 mg/g. However PET waste bottle did not follow this trend, rather it had an initial fast adsorption which rose slightly and decreased sharply.The intraparticle diffusion was significant in the adsorption process of Fe(III) at R^2 values greater than 0.9 for all the single and composite materials except for PET/CHITIN/CLAY with R^2 of 0.8886; however, it was not the controlling step of the adsorption because it was multi-linear. Fe (III) ion adsorption active sites were located in the hydroxyl and C=O functional groups, according to FTIR spectroscopic analysis, Adsorption capabilities, kinetic and bench physicochemical characterization all point to the investigated composites as efficient, eco-friendly, and effective adsorbents for removing Fe (III) ions from solutions.The porosity of waste plastic bottles can be improved by bio-modifiers, and the composites can be used for pollutant remediation.

Conflict of interest

There is no conflict of interest between the authors

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