Removal of phosphorus from sewage effluent by adsorption on Laterite

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Abstract

The present study deals with the removal of phosphorus from wastewater by using laterite as adsorbent, that was collected from Kolhapur. In experiment the effect of contact time, pH , optimum dosege Adsorption isotherm, and Break through curve were studied. The results showed that the optimum dose found to be 15 mg/l , It is observed that removal efficiency of phosphorus increases with decrease in pH of solution. Batch kinetics experiments showed that optimum contact time was about 120 minute. Adsorption capacity was plotted against equilibrium concentration, and isotherm models (Freundlich, Langmuir) were used to correlate these results so it is observed that Langmuir isotherm is fit for adsorption of phosphorus. In the fixed bed isothermal adsorption column, particle size 2 mm and 4 mm, influent flow rate (Q) 10 ml/minute, and bed depth (H) 25 cm were studied here break through curve of 4 mm partical size is slightly deepar than 2 mm partical size. The results showed that the laterite was effective in adsorbing phosphorus, and percent removal of phosphorus reaches up to 89 %

1. Introduction

Phosphorus is contributed to domestic sewage from food residues containing phosphorus and their breakdown products. The use of increased quantities of synthetic detergents adds substantially to the phosphorus content of sewage. Typical total phosphorus concentrations of raw wastewater range from 2 to 20 mg/L. Processes available for P-treatment are categories in to chemical, physical, or biological based treatment systems. Physico-chemical
methods which includes ion exchange, membrane filtration which is used either by conjunction with coagulation or alone. Biological method may not be appropriate for small scale industry because it increases cost and time.

One of the most famous method i.e. adsorption is now a days used as removal or recovery of valuable product as it is low cost, easy to handling and it consumes less reagents. Laterite is one of the adsorbent used as removal of heavy metal i.e. arsenic,

chromium from wastewater. Early observations shown that laterite is a weathering product of the underlying parent rock. The laterite is used for the construction of simple houses and as a road building
material. The suitability of lateritic materials above all of lateritic gravel is tested by several methods of engineering geology. Laterite substrate is rich in Iron (Fe), Aluminium (Al), and Calcium which enhances phosphorus removal. Studies have shown that using substrate rich in iron, aluminum and calcium concentration enhance phosphate removal i.e. Alum Sludge, recovered ochre, black cotton soil, red mud and clay zeolite, bauxite, laterite, dolomite, shale, limestone, calcite, vermiculite and iron-rich sands are naturally occurring minerals tested for phosphorus removal. Some of these are iron and or aluminum rich and some of these are calcium rich. Phosphorus uptake by different local available soil was studied in India (Kothandaraman 2008), and it was investigated that the laterite material had the highest uptake than the soils. Laterite contains high proportions of iron and aluminium oxides, and it is these compounds that allow phosphorous to readily adsorb, and be removed from the water column. The mechanism for this phosphorous adsorption is through a process called ligand exchange. A ligand is a molecule which is bound to a metal ion to form a complex ion. Ligand exchange occurs when the ligand bound to the metal ion is replaced by another ligand. In this case, the orthophosphate molecule acts as a ligand, binding at the hydrous oxides of iron and aluminium, replacing the current ligands, the hydroxides. There is a strong positive correlation between iron- and aluminium oxide content within a soil and phosphorous adsorption.

2.Material and Methods

Analysis of sewage effluent:

Sewage effluent samples were collected in jayantinala from Kolhapur. And analyze physico-chemical parameter like pH, COD, BOD, TSS, TDS, chloride and phosphorus. The parameter were analyzed in the Department of Technology Shivaji University, Kolhapur.

3. Procedure for calculating calibration curve of phosphate:

The first step in the Stannous Chloride method for determining phosphate concentrations was to develop the standard curve using six working standards. The standard curve could then be used with values of absorbance measured by the spectrophotometer in order to analyze samples of unknown phosphate concentration.

Working Standards:

The development of a standard phosphate curve was necessary to establish a relationship between absorbance and phosphate concentration for the spectrophotometric analysis. First, a stock phosphate solution prepared by dissolving 0.7111 g anhydrous KH2PO4 and dilute to 1L; so the 1ml = 0.5 mg PO_4 or 500 mg PO_4/L. and working phosphate solution is reappeared by diluting 100 ml of stock solution to 1lit : 1ml = 0.05 mg PO_4 or 50 mg PO_4/L. A 1L beaker was filled with a standard phosphate solution of which 1.0 ml contained 0.5 mg - P. To each of six 100-ml flasks, the following volumes of the standard phosphate solution were added using pipette and then diluted to the 100 - ml mark: 0.06 -ml, 0.12 -ml, 0.18 -ml, 0.24- ml. 0.30-ml, 0.36 -ml, 0.42 -ml, and 0.48 -ml. These diluted mixtures thus contained 0.3, 0.6, 0.9, 1.2, 1.5 and 2.4 mg- P/L, respectively, and were used as working standards for the analysis. For blank distilled water is used. Colour development is done by adding 4 ml of molybdate reagent and 0.5 ml of stannous chloride reagent –I. Rate of color development and intensity of color depends on temperature of the final solution. Each 1^0 C increase in temperature produce about 1% increase in color. Hence, hold sample, standards, and reagent within 2^0 C of one another and temperature range between 20^0 C and 30^0 C. Color measurement is take place after 10 min, but before 12 min, measure absorbance of the color spectrometrically at 690 nm and compare with a calibration curve using a distilled water as blank.

Fig.1.Standard curve of Phosphate.
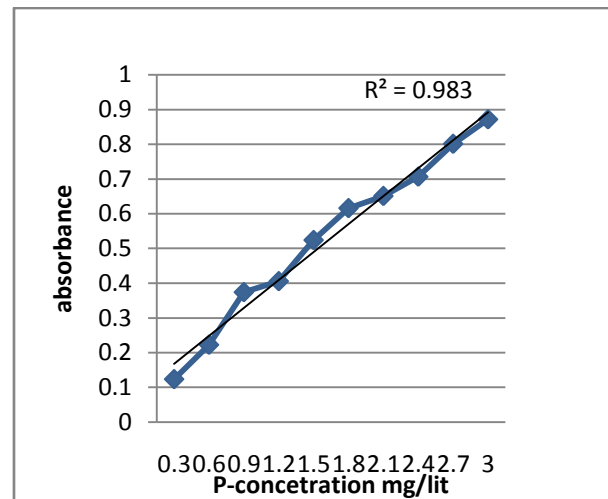
4. Sample collection and Preparation:

The laterite samples were collected from Kolhapur district of Maharashtra. The samples were thoroughly washed in tap water so as to remove all dirt and impurity. They were dried for one day, at 105°C in an oven to remove moisture. Then it was crushed to make adsorbent in different size ranges 0.150 mm, 0.160 mm, 2 mm and 4 mm. These were then sieved, washed thoroughly with large volume of tap water and finally with distilled water. Lastly material was dried in hot air oven at 100^0C for overnight. The as prepared samples were used for phosphate adsorption. The preparation procedure for the adsorbent and the removal of phosphate from aqueous medium is shown in the flow chart (fig 5). Based on Batch studies the particle chosen for phosphate adsorption was of 0.150 mm size. Laterite for phosphate adsorption
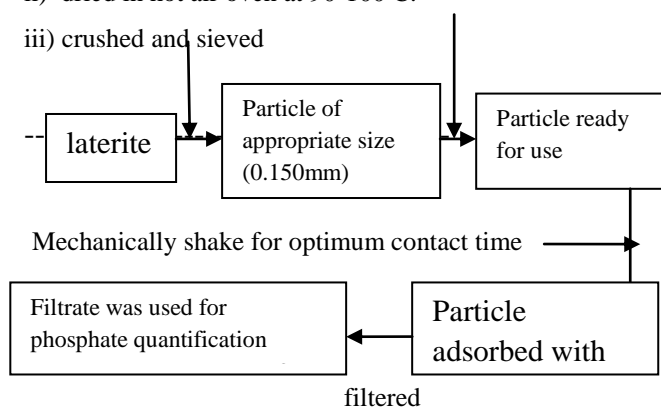
Fig.2. Laterite sample size 0.150 mm

A synthetic phosphorus solution comprising distilled water and potassium dihydrogen phosphate was used in this study. A stock solution of 100 mg / dm^3 was prepared by dissolving a weighed amount of phosphorus in 1000 ml distilled water. The experimental solution was prepared by

diluting the stock solution with distilled water when necessary.

i) washed with distilled water   ii) Dried in sun

ii) dried in hot air oven at 90-100℃.

iii) crushed and sieved

laterite → Particle of appropriate size (0.150mm) → Particle ready for use

Mechanically shake for optimum contact time →

Filtrate was used for phosphate quantification ← Particle adsorbed with

filtered

fig.3. flow chart of the preparation procedure for the adsorbent and removal of phosphate

5.Preparation of artificial wastewater:

The distilled water was spiked with KH_2PO_4 to prepare a phosphate solution of (10 mg/lit) these was used for adsorption studies, and the artificial waste water for column experiment was prepared by adding stock phosphate solution to tap water.

A: Batch experiment: phosphorus removal affinity of laterite determined from batch experiments as a function of contact time, optimum dose of the adsorbent, effect of pH, adsorption isotherms were drawn to these results.

6.Contact time: The adsorption is strongly influenced by the contact time, The equilibrium studies were carried out for phosphate by using laterite concentration of 10 g/L, and the particle size of 150 mm. for equilibrium study 1L of 10 mg /L of phosphate solution mixed with 10 g of laterite. The shaking speed was 168 rpm and the shaking time was varied from 0 to 5 h. The pH of solution was 5.7 ± 0.2. After each hour sample was collected filtered through whatman filter paper and analyzed the remaining P concentration.

7.Determination of optimum dosage of adsorbent:

To determine the optimum dosage of laterite were added to the 1000 ml beaker containing known concentration of phosphate (10 mg/L). The solution in the beaker was subjected to stirring for optimum contact time, sample was taken from beaker filtered and analyzed for residual or removal of phosphate concentration. The dose which gives

minimum residual concentration is chosen as optimum dosage.

8.Effect of pH on phosphorus removal:

The extent of adsorption is strongly influenced by the pH at which adsorption is carried out. The effect of pH on phosphate adsorption was studied by performing equilibrium adsorption test at different pH. The pH of solution was adjusted by using 0.1M NaOH and HCL, by using different pH level to find effect of pH on adsorption of phosphorus. The pH which gives minimum residual concentration is selected as optimum pH.

9.Adsorption isotherm experiment:

The adsorption isotherm curve obtained by plotting the weight of the solute adsorbed per unit weight of adsorbent (qe). Against equilibrium concentration of solute (Ce). The obtained data correlated with Langmuer –Frendalich isotherm model. The parameters for each model were obtained from non linear statistical fit of the equation to the experimental data. All parameters were studied for their correlation coefficients (r2).

B: Fixed bed experiment

Fixed bed experiment includes break through curves for system of different particle size, Adsorption column – The fixed bed adsorption studies were carried out in column of 3.5.cm dia. and 25 cm in height. Screen placed at bottom, and glass
beads at top of the bed to ensure a uniform distribution of influent through laterite.

Fig: 4 : design of column

Inlet

25cm   3.5 cm

Outlet
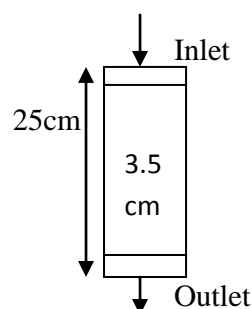
Break through curves for the system by column experiment:

Break through curves for the system for different particle size (fine particle and core particle size) obtained at constant initial concentration of phosphorus and fix bed size, and constant flow rate.

Results and Discussion

10. Analysis of sewage effluent:

Sewage effluent samples were collected in Jayantinala from Kolhapur. The samples were collected for one week, and analyzed for physico-chemical parameters like pH, COD, BOD, TSS, TDS, chloride and phosphorus. The parameter were analyzed in the department of Technology, Shivaji University Kolhapur. The physical characteristics of the Sewage are as shown in table

**Table . 3 . The characteristics of sewage effluent**

Sr.no	Parameter	Range
1	P^H	6.3 - 6.7
2	COD	1700 - 2350
3	BOD	160 - 170
4	TSS	900 - 1100
5	TDS	600 - 850
6	Chloride	70 - 85
7	Phosphorus (ortho)	5 - 15

All fig. Are expressed in mg /l except P^H

11. Effect of contact time:

Contact time has great influence on the adsorption process. Effect of contact time studies were conducted separately for phosphate to find out the equilibrium time for phosphate uptake by laterite. In this study initial concentration of phosphate was taken as 10 mg/L, adsorbent doses were taken as 10 g/L for phosphate respectively, and the adsorbent size was 0.150 mm. The pH of the solution was 5.7 ± 0.2 and the shaking time varied from 0 – 5 hours, at an agitation speed of 168 rpm

Fig 4 Conditions : P concentration = 10 mg/l, temp = 29^0C ± 2^0 C, absorbent dose = 10 g/L, agitation speed = 168 rpm, p^H = 5.7 ± 0.2 .
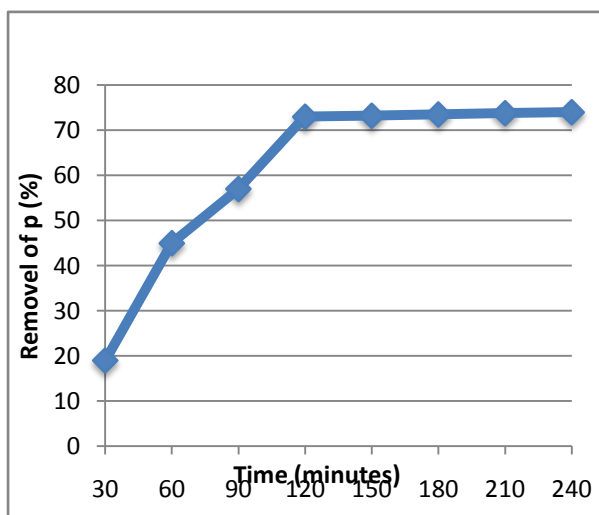
It was noticed that most of the adsorption (~ 51% for phosphate) occurs in the first 1 hour of shaking. Further shaking of 3 hours caused ~73% removal of phosphate (Fig.4). Thus a contact time of 120 minute was chosen for the attainment of the equilibrium for phosphate. The table 4 shows that the time and P - remaining after successive time period. and table 5 shows the time period and % removal of phosphate.

12. Effect of pH:

The pH value of the aqueous solution is an important controlling parameter in the adsorption process. These pH values affect the surface charge of adsorbent, the degree of ionization and speciation of adsorbate during adsorption process. Thus, the effect of pH (H+ ion concentration) in the solutions on the adsorption percentage of phosphate ions was studied at different pH ranging from 2 to 12.

The effect of pH on percent phosphate removal is shown in Figure 9. The reported percent phosphate removals were obtained using a laterite dosage of 15 g/l, an initial phosphate concentration of 10.0 mg/l, and a contact time of 120 min. The percentage of phosphate ions bound by laterite was determined from the ratio of phosphate ions present in the solution as given by Equation 1:
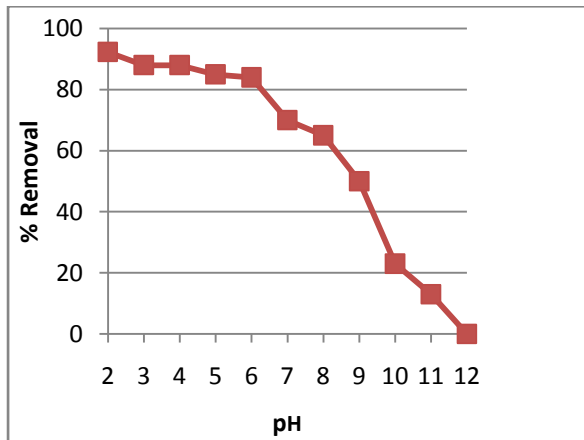
$$\% \text{ Phosphate removal} = \left(\frac{C_o - C_e}{C_o} \right) * 100\%$$

where,

C_o = initial phosphate concentrations mg/l.

C_e = equilibrium phosphate concentrations mg/l.

Fig: 5 : Conditions; concentration 10 mg/l, temp $\pm 29^0$C, Adsorbent dose =15 mg/l, agitation speed = 168 rpm, contact time 120 minute.

Table no . 7

Sr.no	Time in (hr)	conc. mg/l	C/co
1	1	0.5	0.05
2	3	0.6	0.06
3	4	0.8	0.08
4	24	4.79	0.47
5	27	4.48	0.48
6	28	5.64	0.56
7	52	6.74	0.67
8	76	7	0.7
9	100	8	0.8
10	124	10	1

C = equilibrium phosphate concentrations mg/l
Co = initial phosphate concentrations mg/l

Fig. 6 . Condition: P - conc.10 mg/l, flow rate 10 ml/ minute, bed depth 25 cm, particle size 2 mm.

The results obtained indicate that adsorption of phosphate on laterite increases with decreasing pH, with the highest adsorption capacity (93.02 %) obtained at pH 2 ± 0.10 (the lowest pH used in this investigation). The amount of phosphate ions removed from solution by at equilibrium pH 2 ± 0.10 (93.02%), was more than that removed by laterite at the equilibrium pH of 10.03 ± 0.10 (53%). This observation can be attributed to the effects of solution pH on phosphate speciation and charge development on the laterite surface

13.Effect of Particle Size:

Column experiment

Two column experiments were carried out at different particle size (2 mm and 4 mm), see Table 7 and 8. Columns with an inner diameter of about 3.5 cm were made of PVC column and placed with their axis in the vertical direction. The columns were filled with the laterite a fixed bed and a synthetic solution of PO_4-P influent (KH_2PO_4 dissolved in tap water) was driven from the top of the columns downward direction. The experiments were carried out at room temperature (29°C). Effluent samples were taken twice daily from each container and analyzed with respect to dissolved phosphorus (about 100 mL filtrated through 0.45 μm filters), PO_4-P breakthrough curves obtained from column experiments were used in this study.

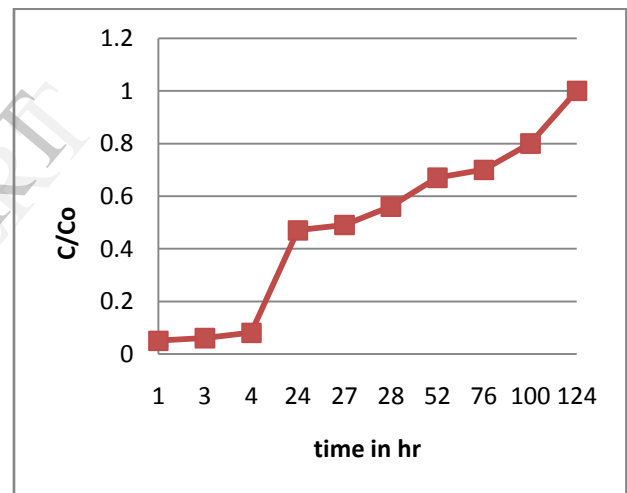## 14.Breakthrough curves for particle of size 2 mm.:

The breakthrough curve as shown in fig.6 was obtained from particle of size 2 mm and at constant initional concentration of phosphorus (10 mg/L), the bed depth of laterite was 25 cm, and at constant flow rate (10 ml/minute). the experimental result in table 7 show that time in hour, C/Co, and concentration of phosphate after successive time,.

## 15.Breakthrough curves for particle of size 4 mm:

The breakthrough curve as shown in fig..7. was obtained from partical of size 4 mm and at constant initional concentration of phosphorus (10 mg/L), the bed depth of laterite was 25 cm, and at constant flow rate (10 ml/minute). the experimental result in table 8 shows that time in hour, C/Co, and concentration of phosphate after successive time.

Table no : 8

Sr.no	Time in (hr)	Conc. mg/l	C/co
1	1	0.5	0.05

2	3	0.6	0.06
3	4	0.7	0.07
4	24	4.2	0.42
5	27	4.23	0.423
6	28	5.01	0.501
7	52	6	0.6
8	76	6.99	0.699
9	100	7.99	0.799
10	124	10	1

Fig .7 . Condition p - conc.10 mg/l, flow rate 10 ml/ minute, bed depth 25 cm, particle size 4 mm.
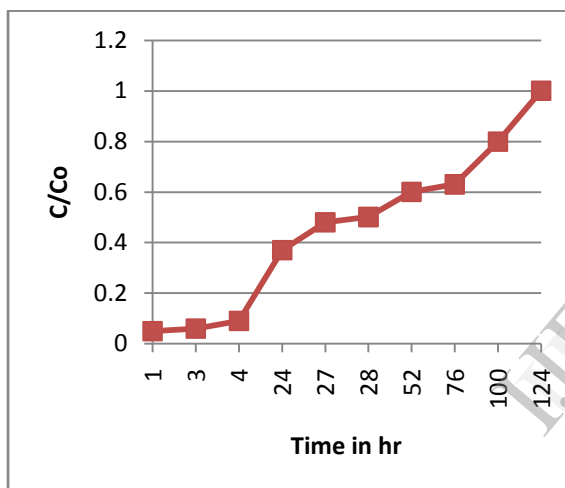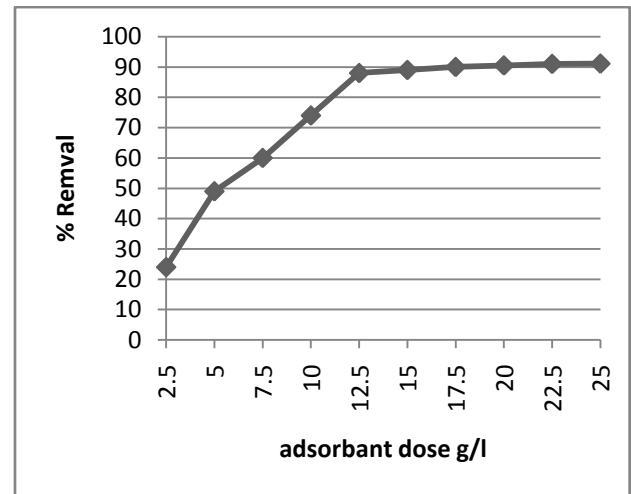

16.Adsorption isotherm:
Effect of adsorbent dose:

The effect of the adsorbent dose was studied at room temperature (29 - 30°C) by varying the sorbent amounts from 2.5 to 25 g/L. For all these runs, initial concentration of phosphate was fixed as 10 mg/L. The agitation speed was 168 rpm and the contact time was 120 minute. and at pH 5.23 - 5.56. Figure 8 shows the adsorption of phosphate increases rapidly with increase in the amount of laterite due to greater availability of the surface area at higher concentration of the adsorbent. The significant increase in uptake was observed when the dose was increased from 5 to 15 g/L. Any further addition of the adsorbent beyond this did not cause any significant change in the adsorption. This may be due to overlapping of adsorption sites as a result of overcrowding of adsorbent particles . It is observed that ~89 % removal for phosphate with 15 g/L of adsorbent dose.

Fig. 8. Conditions; concentration 10 mg/l, temp +/- 29^{0}C, dose was increased from 5 to 15 g/L.

agitation speed =168 rpm, contact time 120 minute. P^{H} 5.23 -5.56.

17.The Langmuir isotherm:

The Langmuir model is based on the assumption that a fixed number of accessible site are available on the adsorbent surface, all which have same energy,and second is adsorption is reversible . Equilibrium is reached when the rate of adsorption of molecules onto the surface is the same as the rate of desorption of molecule from the surface.
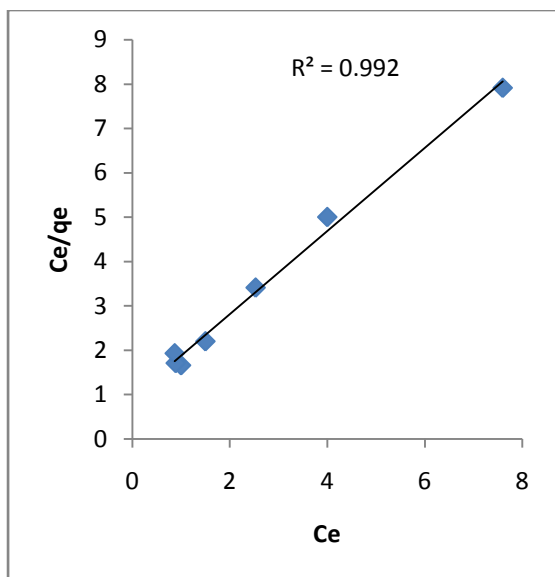
The Langmuir isotherm is given by:

$$qe = \frac{qe\, KL\, Ce}{1 + KL\, Ce}$$

The constants in the Langmuir isotherm can be determined by plotting (1/qe) versus (1/ Ce) and making use of above equation rewritten as:

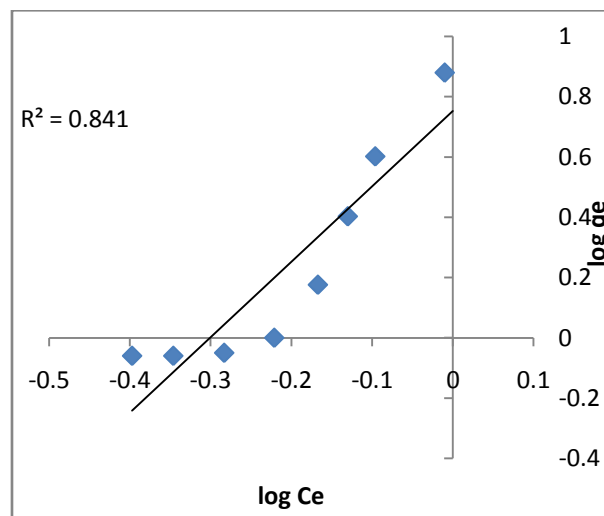$$\frac{1}{qe} = \frac{1}{qm} + \frac{1}{qm\, KL}\cdot\frac{1}{Ce}$$

where *qm* and *KL* are the Langmuir constants. It can be seen from Fig. 9 that the isotherm data fits the Langmuir equation well ($R^2 = 0.992$). The values of *qm* and *KL* were determined from the figure and were found to be 1.32 mg/g and 0.83 L/mg, respectively

Fig. 9. Langmuir Adsorption Isotherm.

18.The Freundlich isotherm:

The Freundlich isotherm model is an empirical relationship describing the adsorption of solutes from a liquid to a solid surface and assumes that different sites with several adsorption energies are involved. Freundlich adsorption isotherm is the relationship between the amounts of phosphate adsorbed per unit mass of adsorbent, qe, and the concentration of the phosphate at equilibrium, Ce.

$$qe = K f \, Ce^{1/f}$$

The logarithmic form of the equation becomes,

$$Log\ qe = \ log\ K f\ + \ \frac{1}{n}\ log\ Ce$$

where Kf and n are the Freundlich constants, the characteristics of the system. Kf and n are the indicators of the adsorption capacity and adsorption intensity, respectively. The ability of Freundlich model to fit the experimental data was examined. For this case, the plot of log Ce vs. log qe was employed to generate the intercept value of Kf and the slope of n. From Fig. 10 the Freundlich constants Kf and1/ n were found to be 0.50 and 0.4 respectively. The magnitudes of Kf and n show easy separation of phosphate ions from the aqueous solution and indicate favourable adsorption. The intercept Kf value is an indication of the adsorption capacity of the adsorbent; the slope 1/n indicates the effect of concentration on the adsorption capacity and represents adsorption intensity. The Freundlich isotherm is more widely used but provides no information on the monolayer adsorption capacity in contrast to the Langmuir

model. Freundlich isotherm fitted well with the correlation coefficient of 0.841.

Fig . 10 . The Freundlich isotherm.

Isotherm Models Constants and Correlation Coefficients for Adsorption of phosphate from aqueous solution.

Adsorbent	q_m(mg/g)	K_L (L/mg)	R^2
Laterite	1.32	0.83	0.992

Freundlich isotherm

Adsorbent	K_f	1/n	R^2
Laterite	0.50	0.40	0.84

19.Summary and Conclusion:

Adsorption studies were conducted to investigate the adsorption capacity of laterite under different conditions based on the result of their studies, the laterite can be used as an adsorbent for the removal of phosphate from synthetic sewage effluent. Laterite was easily available in large quantity and the treatment method of adsorption seemed to be economical. The amount of phosphate adsorbed was found vary with p^H of solution, adsorption dose, particle size of laterite, contact time.

Following are the conclusions from the present study.

- The present study shows that the laterite can be successfully used for the removal of phosphate from sewage effluent.
- The laterite proved to be an effective adsorbent and removal capacity was found around 89 %.
- It was found that percentage removal efficiency of phosphorus depends on adsorbent dose and size of the adsorbent material. The phosphorus removal

increases with adsorbent dose. As such phosphorus removal efficiency increases with decreasing size of the adsorbent material.

- Study of the adsorption as function of contact time showed that 120 minute was sufficient time to maximum removal of phosphate.

- There is no need for the pretreatment of the laterite with any chemicals such as acid or base. This minimizes the cost of adsorbent preparation

- pH value is an important factor affecting the adsorption of phosphate on laterite. Acidic environment (pH = 2 – 5) is favorable to the adsorption of phosphate. the amount of adsorption decreases with increasing pH value of the solution.

- In fixed bed experiment, Exhaustion point of break through curve was found after 124 hr for 2 mm partical of laterite. and 123 hr for 4 mm partical of laterite

- In adsorption isotherms experiment Freundlich model was fit to the adsorption. Taking into consideration of the above results, it can be concluded that the laterite is a suitable adsorbent for the removal of phosphate ions from wastewater in terms of low cost, abundant availability.

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