



Investigating the Efficiency of Lightweight Expanded Clay Aggregate (LECA) in Wastewater Treatment of Dairy Industry

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ABSTRACT: Lightweight expanded clay aggregate due to its physical and chemical properties; it can increase the process of adsorption and ion exchange. In this study, the efficiency 2 types of Lightweight Expanded Clay Aggregate in reducing the amount of COD, BOD, TSS, nitrate and phosphate in dairy industry wastewater were investigated and the impact of different factors such as adsorbent dosage, mixing speed and mixing time were studied. The results showed that the efficiency of COD reduction of wastewater after touch with the granular type after 20 h is 65.9%. TSS and BOD have also been greatly reduced. Also the use of powder type, the highest removal efficiency was 31.81%, mixing speed 100 rpm, mixing time 20h, and adsorbent dosage 10 gr/l in addition; increasing the rate of mixing speed increases the amount of nitrate absorption. The most effective nitrate removal is 63.87% which was at mixed speed 200 rpm and adsorbent dose 4 g/l. Results show that, the mixing speed has little effect on the absorption of phosphate. So, after 30 minutes, even with increasing mixing speed for doses 2 & 4 g/l, the reduction efficiency also decreases the change in adsorbent dose from 1 g/l to 4 g/l was virtually unaffected. Increased mixing speed is due to better distribution of nitrate and phosphate molecules in the solution and their contact with adsorbent increased the absorption rate. According to the results, the LECA granular has more ability to remove the COD.

Keywords: Lightweight expanded clay aggregate (LECA), dairy wastewater, COD, nitrate, phosphate

INTRODUCTION

The development of various industries has had many positive and negative impacts. With the advancement of technology and increase of various industries, wastewater from these industries is a serious threat to the environment. Factories and industrial units according to their activity daily a large amount of industrial health and wastewater impose into the environment. Among the industries, the sewage has a high organic load is sewage dairy industry (Hamedian, 2000). The biodegradability of the wastewaters, biological methods, like the stabilization pond, activated sludge, anaerobic up-flow reactor (UASB) and sequential batch reactor (SBR) is used for treating them. The main problems common sewage treatment systems can be high costs of construction, high energy consumption, the need for complex operation and requires treatment and disposal of sludge and noted that the use of mechanized systems mainly use high-end technology. Among all wastewater treatment methods, absorption due to low cost and

ease of operation is considered as one of the best methods. Many natural ingredients such as fruits, sawdust and other wood materials, seaweed and algae, peat moss covered Zeolite and other pollutants are used as adsorbent (Imran et al, 2012). This method compared to other methods that are used to remove various contaminants from the advantages of being cost-effective, selective absorption capability, the ability to regenerate and recover metals has high relative speed of the process and lack of sludge production (Naddafi et al, 2005). One of the materials that has recently been considered in the sewage treatment process is Lightweight expanded clay aggregate (LECA). LECA is a volcanic rock that is found in most parts of the world including Iran. LECA is usually due to high porosity, lighter floats on the water surface (Zarabi et al, 2011). LECA or expanded clay is a light weight aggregate made by heating clay to around 1,200 °C (2,190 °F) in a rotary kiln (Tor Arne et al, 2000). The emitted gases are blocked in pores due to a high viscosity of the liquid phase. The

liquid phase blocked in the pores causes the expansion of granules (Ehlers 1958; Latosinska & Zygadlo 2009). Much of this mineral of aluminum oxide (AlO_2) and silicon oxide (SiO_2) is formed and a small percentage of these minerals include oxides of sodium, potassium, iron and magnesium (Esmaili et al, 2004). The main mineral of aluminum oxide (AlO_2) and silicon oxide (SiO_2) is formed and a small percentage of these minerals include oxides of sodium, potassium, iron and magnesium (Latosinska & Zygadlo 2009; Esmaili et al, 2004). A special blend of LECA and efficiency of adsorbents about the absorption process and ion exchange raises, the reason is that materials with a high percentage of Silica are capable of becoming Zeolites that as natural exchangers are frequently used in environmental engineering (Zarabi et al, 2011). In this study, the efficiency LECA in reducing the amount of COD, BOD, TSS, phosphate and nitrate in dairy industry wastewater were investigated and the impact of different factors such as adsorbent dosage, mixing speed, and mixing time were studied. So far, several different studies on the application of LECA or similar adsorbent for the purification of different industrial wastewater are summarized below:

Asantoa et al (2012); Prove that of a kind of LECA used to remove three types of PAHs in wastewater and mixing time, adsorbent dosage and adsorption isotherms studied. The results show that the maximum absorption (optimal absorption) 21 hours after amount of PAHs remained stable. By increasing the amount of adsorbent, increases absorption. The results were consistent with Langmuir and Freundlich isotherm. Esmaili et al (2004); stated that absorption of copper (Cu^{+2}) and nickel (Ni^{+2}) from aqueous solutions by volcanic ash at a temperature of 27 ± 1 as a function of pH, adsorbent dosage, metal concentration, mixing time tested. By increasing the concentration of metal, but reduced absorption per unit increases mass of sorbent. Equilibrium time is very short and it shows the locations available are well absorbed. The study shows that the uptake of copper is more than nickel. Kavosi and Barqae (2005); they claimed that LECA as a biofilm support the use of biological treatment of wastewater containing sugar molasses with nutrients examined. The results highlight that the reactor is capable of removing 82% of soluble input load. In addition. Zarabi et al (2011); they showed that efficiency of LECA improved by Acid Dyes in Hydrochloric filtration of textile effluents was evaluated. Sharifinia et al (2012); they claimed that the uptake of ammonium by a LECA was examined. The results show that they have taken into account the equilibrium constant in all temperature range of

Langmuir model. Other scholars who have studied in this field include: Moradian et. Al (2013); Eikebrokk et al (2001); Casido (2011); Haque et al (2011); Azari et al (2014) and Malakootian et al (2009).

MATERIALS AND METHODS

LECA was prepared of Khajeh Abad mine located in Sarvelayat section in the city of Nishapur in the Khorasan province. LECA by XRF (fluorescence spectrophotometer) was analyzed. Specification and weight percentage of adsorbent is presented in Table 1. The wastewater from a dairy factory was prepared in real and not simulated, in Nishapur. All experiments were carried out at the Laboratory of Islamic Azad University, Shahrood Branch. Experiments were conducted in two phases. In the first phase, LECA was in granular form and in the second phase, in powder form. Also, in the second phase, experiments were carried out in two separate sections: the removal of nitrate and phosphate in the first section and the removal of COD were investigated in the second section. All experiments were repeated 3 times. Figure 1 shows the research steps.

The parameters measured in each phase were:

- Phase 1: percentage / rate of COD, BOD and TSS;
- Phase 2: percentage / rate of COD, (P_3O_4) and (NO_3^-)

For removal of initial impurities, LECA before crushing, as granules were washed with distilled water several times, and was exposed to ambient air until completely dry. Then, by the crushing and grinding samples were milled using standard ASTM sieves with mesh sizes 60-80 particle graded. Thus; 2 sieves with a mesh of 60 and 80 were on each other (mesh 60 at the top and sieve with mesh 80 at the bottom). The LECA powder was passed through a mesh sieve 60 and whatever remained on mesh sieve 80 were collected for tests.

LECA powder collected from the mesh80, washed several times by boiling distilled water to remove inorganic salts that are soluble and sticking to the surface and turbidity of the effluent is reduced as much as possible. After using of filter paper and vacuum pump isolated from effluent was washed, dried casings. Then powder after filtration LECA for 3 hours in an oven at 120°C was placed to dry completely. After complete drying in desiccators, and then cooled to ambient temperature. In the following, all samples after the desired time, they were first passed through filter paper and then placed in a centrifuge machine (For 5 m and 6000 rpm). The paper was filtered again and then the nitrate and phosphate levels were measured

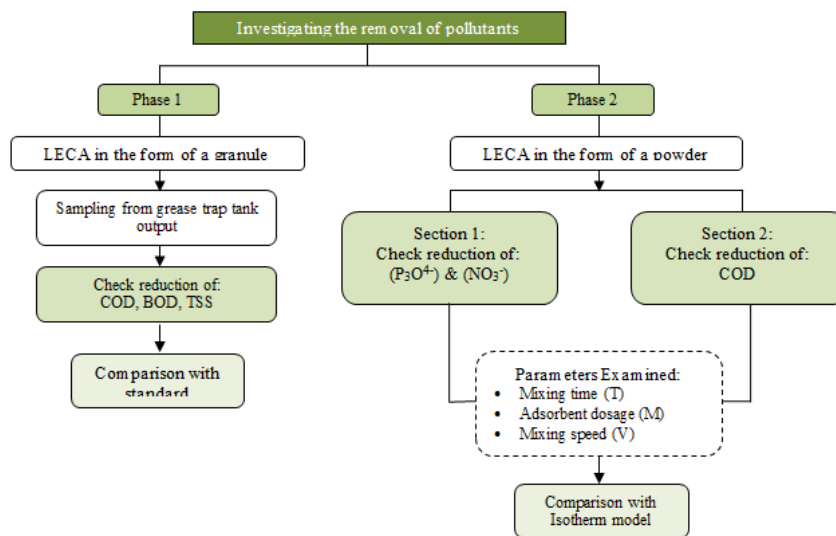


Figure 1. Flowchart of steps and components of research

Table 1. Specification and weight percentage of adsorbent (LECA)

Sample	SiO ₂ %	Al ₂ O ₃ %	Na ₂ O %	MgO %	K ₂ O %	Ti ₂ O %	MnO %	CaO %	P ₂ O ₅ %	Fe ₂ O ₃ %	SO ₃ %	LOI %
40281-H	50.74	16.35	3.04	3.31	0.67	1.11	0.12	9.46	0.40	11.73	0.00	3.08
Sample	Ba ppm	Co ppm	Cr ppm	Cu ppm	Nb ppm	Ni ppm	U ppm	Th ppm	Ce ppm	Cl ppm	Pb ppm	Rb ppm
40281-H	338	35	180	20	11	147	N	31	62	270	48	24
Sample	Sr ppm	V ppm	Y ppm	Zr ppm	Zn ppm	Mo ppm						
40281-H	862	180	15	225	67	12						

In this study, three parameters (operating) mixing time (T), adsorbent dosage (M) and mixing speed (V) were studied. Based on the literature review (Mc Graw – Hill, 2000) and preliminary tests, for the mixing time 3 levels, for the adsorbent dosage 3 levels, and for mixing speed 2 levels were considered. Initial pH of sewage was 8. Duration 6

hours to reach equilibrium, based on kinetic study was elected. Of course, the range of mixing time and adsorbent dosage are variables in different experiments (COD removal and nitrate and phosphate removal). The parameters and levels are shown in Table 2 & 3.

Table 2. Parameters and levels considered for nitrate & phosphate removal

Parameter	Level 1	Level 2	Level 3
Mixing time (min)	30	60	120
Adsorbent dosage (g/l)	1	2	4
Mixing speed (rpm)	100	200	-

Table 3. Parameters and levels considered for COD removal

Parameter	Level 1	Level 2	Level 3
Mixing time (h)	16	20	24
Adsorbent dosage (g/l)	1	5	10
Mixing speed (rpm)	100	200	-

Table 4, shows the designed experiments based on the parameters and levels (For each phases individually). In order to increase the accuracy and reduce the error rate, all variables and possible states were tested. Reproducibility was performed by 2 times and average the results of the analysis were used.

Table 4. Designed experiments based on the parameters and levels

Test No.	Mixing time	Adsorbent dosage	Mixing speed
1	t ₁	M ₁	V ₁ (100)
2	t ₁	M ₁	V ₂ (200)
3	t ₁	M ₂	V ₁
4	t ₁	M ₂	V ₂
5	t ₁	M ₃	V ₁
6	t ₁	M ₃	V ₂
7	t ₂	M ₁	V ₁
8	t ₂	M ₁	V ₂
9	t ₂	M ₂	V ₁
10	t ₂	M ₂	V ₂
11	t ₂	M ₃	V ₁
12	t ₂	M ₃	V ₂
13	t ₃	M ₁	V ₁
14	t ₃	M ₁	V ₂
15	t ₃	M ₂	V ₁
16	t ₃	M ₂	V ₂
17	t ₃	M ₃	V ₁
18	t ₃	M ₃	V ₂

COD measurement in this study using reflux is closed. For this purpose, boiling stones added Erlenmeyer flask 250ml or 500ml and 20cc sample with pipette added to Erlenmeyer flask then 20cc dichromate normal 0.25 added and mixed. Then 5cc concentrated sulfuric acid 98% added and on the tip of the spatula, silver nitrate was added. Then, Erlenmeyer flask is connected to the refrigerant and cooling water flow in the refrigerant. To ensure that the steam outlet covered the upper portion of refrigerants with a small Erlenmeyer flask and sample is returned to reflux for 2 hours. The entire above step is done on 20cc distilled water as a control. After 2 hours, heat cut and without releasing refrigerant allows to sample cooled. Then 80cc distilled water is added from the top of refrigerants. Erlens separated from the refrigerant after cooling completely, add 5 drops of Freon into each Erlenmeyer flask and under Burt containing ammonium sulfate until the appearance of reddish brown color prepared.

Ferrous Ammonium Sulfate ((NH₄)₂Fe (SO₄)₂•6H₂O) consumption for each flask notes the following in relation is (Kargi & Uygur 2003; Mary Selecky 2005).

$$(1) \quad COD \frac{mg}{L} O_2 = \left(\frac{(A-B) \times N \times E \times 1000}{V} \right)$$

As follows:

- A: volume of ferrous sulfate is used is control and in this study were obtained 20cc value
- B: Sample consumption of ferrous sulfate per ml
- N: normality of ferrous sulfate modification
- E: equivalent is the amount of which 8gr of oxygen
- V: volume of the sample in terms of cc

RESULTS AND DISCUSSION

Results of phase 1: LECA in the form of a granule

As it mentioned, in the study the LECA was used in its natural (granules) and results in the table provided below.

Table 5. results of the application of LECA in the form of granules on dairy wastewater

Parameter	The main sample after grease trap tank / fairly raw wastewater (mg/l)	sample after touch with the LECA for 20h	Removal efficiency
COD	2200	750	65.9%
BOD	1220	385	68.4%
TSS	570	225	60.5%

As it is observed, the efficiency of COD reduction of wastewater after touch with the granular LECA after 20h is 65.9%. TSS and BOD have also been greatly reduced. A comparative comparison of the results and the standards presented in the figure 2 is shown.

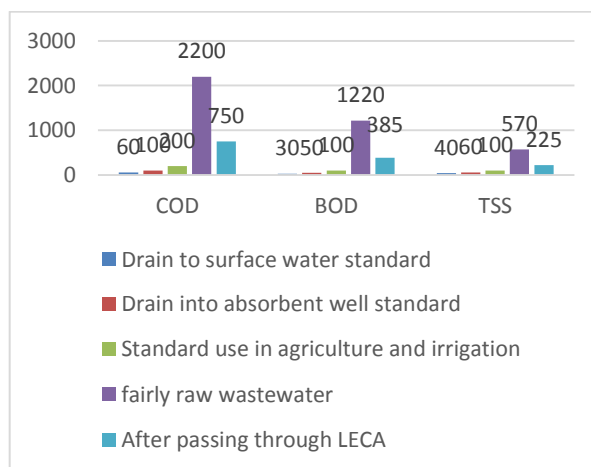


Figure 2. Compare initial output and waste passed from LECA granules with standards Department of Environment (2006) / based on (mg/l)

As is clear, although all the parameters have been greatly reduced, they are still far from national standards about wastewater treatment.

Results of phase 2: LECA in the form of powder

The results are presented in 2 sections:

◆ **Section 1: COD removal:** To determine the Removal efficiency of COD, Initially, the COD level of the fairly raw wastewater (grease trap tank output) was measured. Then exposed to LECA in powder form for 20h and after that Re-measured. The study results are presented in the table 6.

Table 6. results of the application of LECA in powder form on the dairy wastewater

Test no.	Mixing time (h)	adsorbent dosage (g/l)	Mixing speed (rpm)	COD main sample / fairly raw wastewater	sample after passing from LECA for 20h	Removal efficiency (%)
1	16	1	100	2640	2100	20.45
2	16	1	200	2640	2460	6.81
3	16	5	100	2640	1980	25
4	16	5	200	2640	2340	11.36
5	16	10	100	2640	1920	27.27
6	16	10	200	2640	2280	13.63
7	20	1	100	2640	2100	20.45
8	20	1	200	2640	2340	11.36
9	20	5	100	2640	2100	20.45
10	20	5	200	2640	2400	9.09
11	20	10	100	2640	1800	31.81
12	20	10	200	2640	2280	13.63
13	24	1	100	2640	2280	13.63
14	24	1	200	2640	2340	11.36
15	24	5	100	2640	2100	20.45
16	24	5	200	2640	2400	9.09
17	24	10	100	2640	1980	25
18	24	10	200	2640	2160	18.18

According to numbers obtained for the removal of COD, it is observed that the maximum removal efficiency was 31.81% in this experiment, mixing speed 100rpm, mixing time 20h, and adsorbent dosage 10gr/l (Highlighted). On the other hand, at the same mixing time and adsorbent dosage, it seems that with increasing mixing speed, efficiency will be reduced. Thus; by increasing the mixing speed, efficiency (remove COD) is reduced. This means at the same mixing time and adsorbent dosage, optimum mixing speed is 100rpm. In addition, minimum of removal efficiency was 6.81% in this experiment, mixing speed 200rpm, mixing time 16h, and adsorbent dosage 1gr/l.

◆ **Section 2: nitrate and phosphate removal:** The table 7 shows the equilibrium concentrations of nitrate.

In general, increasing the rate of mixing speed increases the amount of nitrate absorption. In the first 30 minutes, for the same adsorbent doses with increasing mixing speed increasing the removal efficiency is also significant. In fact, the efficiency is almost doubled (Figure 3).

As specified in the table, the most effective nitrate removal is 63.87% which was at mixed speed 200 rpm and adsorbent dose 4 g/l. Also, maximum rate of absorption capacity is 22.38 mg/l which was at mixed speed 200 rpm and adsorbent dose 1 g/l. As shown in the figure 4, given the correlation coefficient obtained, and distribution of points it can be said that, Langmuir model is not a suitable model for justifying nitrate uptake and the changes from C_e/q_e to C_e are not linear.

Table 7. equilibrium concentrations of nitrate

Test no.	Mixing time (h)	adsorbent dosage (g/l)	Mixing speed (rpm)	Initial concentration (C_0)	Equilibrium concentration (C_e)	Percent reduction $\frac{C_0 - C_e}{C_0} \times 100$	Absorption capacity $q_e = \frac{(q_e)(C_0 - C_e)V}{M}$
1	30	1	100	50.38	40.28	20	10.1
2	30	1	200	50.38	30.26	39.93	20.12
3	30	2	100	50.38	36.25	28.04	7.06
4	30	2	200	50.38	27.23	45.95	11.57
5	30	4	100	50.38	34.43	31.72	3.98
6	30	4	200	50.38	25.32	49.74	6.26
7	60	1	100	50.38	35.08	30.36	15.3
8	60	1	200	50.38	28.5	43.42	21.88
9	60	2	100	50.38	29.05	42.33	10.66
10	60	2	200	50.38	19.03	62.22	15.67
11	60	4	100	50.38	20.27	59.76	7.52
12	60	4	200	50.38	18.8	62.68	7.89
13	120	1	100	50.38	34	32.51	16.38
14	120	1	200	50.38	28	44.42	22.38
15	120	2	100	50.38	28	44.42	11.19
16	120	2	200	50.38	18.7	62.88	15.84
17	120	4	100	50.38	20	60.3	7.59
18	120	4	200	50.38	18.2	63.87	8.04

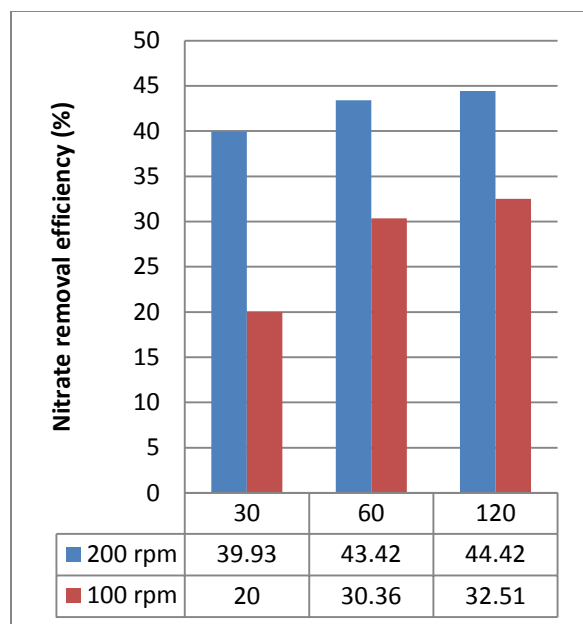


Figure 3. The effect of mixing speed on the efficiency of nitrate removal in the adsorbent dose of 1 g/l and different contact times

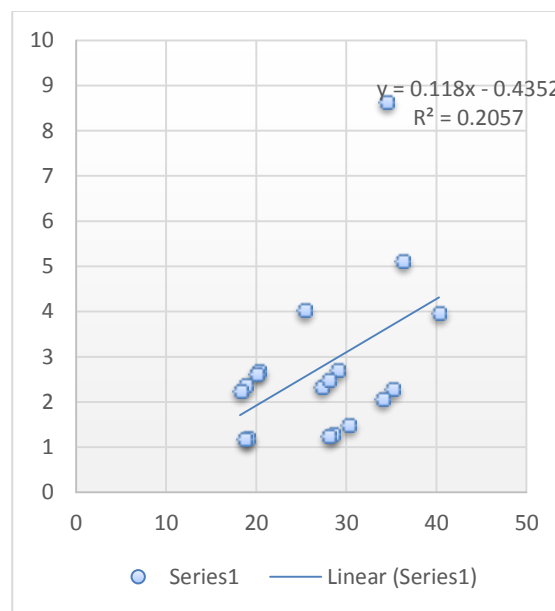


Figure 4. Langmuir isotherm model diagram for nitrate removal

The table 8 shows the equilibrium concentrations of phosphate.

Table 8. equilibrium concentrations of phosphate

Test no.	Mixing time (h)	adsorbent dosage (g/l)	Mixing speed (rpm)	Initial concentration (C ₀)	Equilibrium concentration (C _e)	Percent reduction $\frac{C_0 - C_e}{C_0} \times 100$	Absorption capacity $q_e = \frac{(C_0 - C_e)V}{M}$
1	30	1	100	29.3	14.56	50	14.74
2	30	1	200	29.3	14.48	50.5	14.82
3	30	2	100	29.3	12.30	58.02	8.5
4	30	2	200	29.3	12.1	58.7	8.6
5	30	4	100	29.3	11.72	60	4.39
6	30	4	200	29.3	11	62	4.57
7	60	1	100	29.3	12	59	17.3
8	60	1	200	29.3	10.5	64	18.8
9	60	2	100	29.3	9.6	67	9.85
10	60	2	200	29.3	9.8	66	9.75
11	60	4	100	29.3	9.4	67	4.97
12	60	4	200	29.3	9.48	67.64	4.95
13	120	1	100	29.3	12.2	58.36	17.1
14	120	1	200	29.3	10.48	64.23	18.82
15	120	2	100	29.3	9.58	67.3	9.86
16	120	2	200	29.3	9.7	66.89	9.8
17	120	4	100	29.3	9.4	67.91	4.97
18	120	4	200	29.3	9.44	67.78	4.96

Increasing the mixing speed for same adsorbent doses and at different contact times does not significantly affect the removal efficiency (Figure 5).

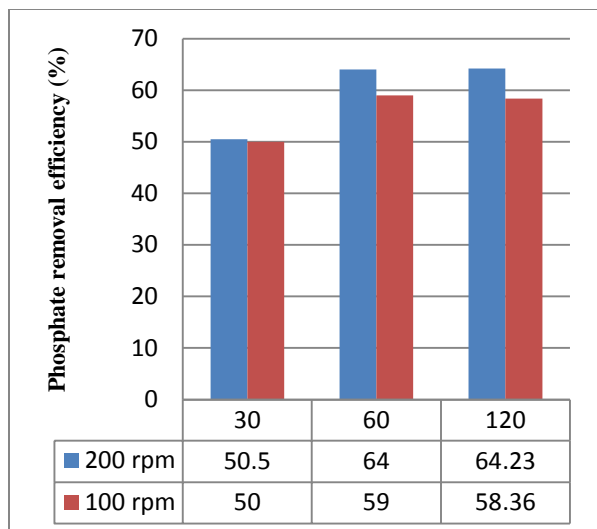


Figure 5. The effect of mixing speed on the efficiency of phosphate removal in the adsorbent dose of 1 g/l and different contact times

Results show that, the mixing speed has little effect on the absorption of phosphate. So, after 30 minutes, even with increasing mixing speed for doses 2 & 4 g/l, the reduction efficiency also decreases. Maximum rate of absorption capacity was at mixed speed 200 rpm and adsorbent dose 1 g/l (like nitrate). As shown in the figure 6, given the correlation coefficient obtained (correlation coefficient is very small), and distribution of points it can be said that, Langmuir model is not a suitable model for justifying phosphate uptake and the changes from C_e/q_e to C_e are not linear.

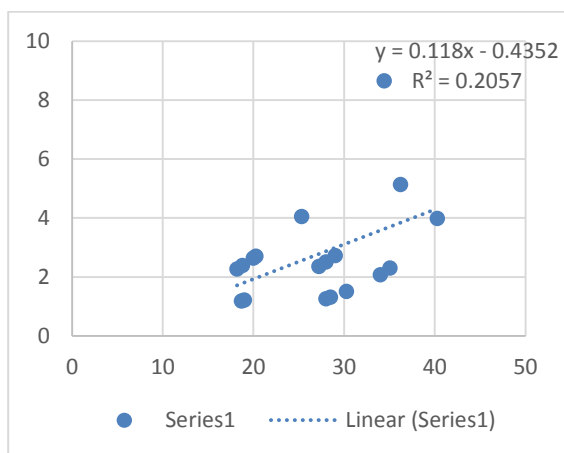


Figure 6. Langmuir isotherm model diagram for phosphate removal

In the following figures, the effect of contact time and adsorbent dose on the removal of nitrate and phosphate is given.

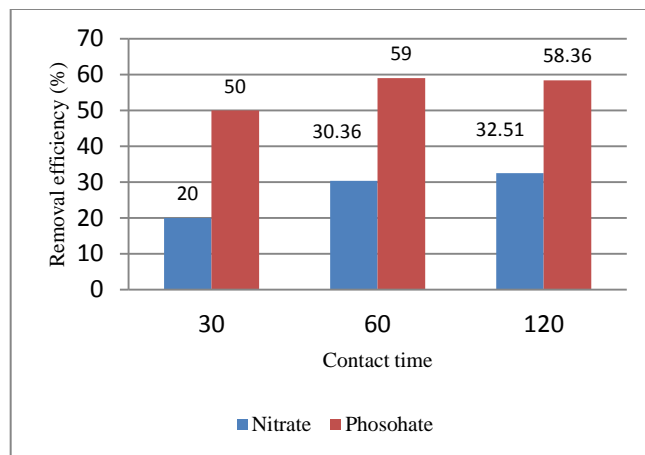


Figure 7a. Effect of contact time on the removal of nitrate and phosphate at adsorbent dose 1 gr/l and mixing speed 100 rpm

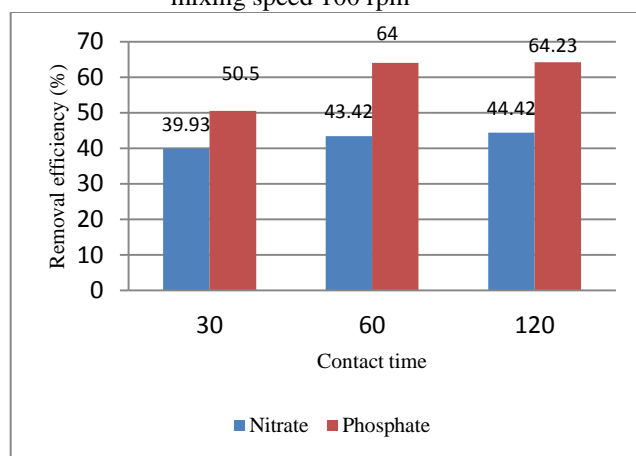


Figure 7b. Effect of contact time on the removal of nitrate and phosphate at adsorbent dose 1 gr/l and mixing speed 200 rpm

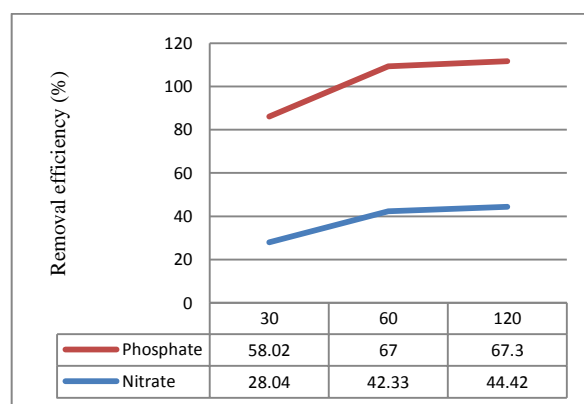


Figure 7c. Effect of contact time on the removal of nitrate and phosphate at adsorbent dose 2 gr/l and mixing speed 100 rpm

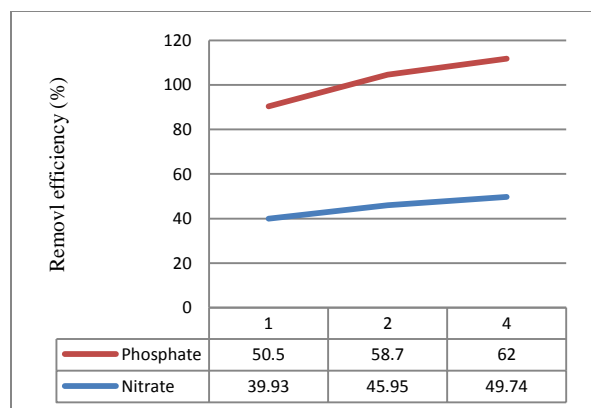


Figure 7d. Effect of contact time on the removal of nitrate and phosphate at adsorbent dose 2 gr/l and mixing speed 200 rpm

Based on the above diagrams, it can be seen that both the mixing speed 100 -200 rpm and the different contact times always have the effect of removing Phosphate more than nitrate.

CONCLUSION

According to the results, with increasing contact time in most experiments, the absorption rate increased that. The reason for this is an increase in the probability of contact with the absorbent surface and the highest amount of nitrate and phosphate uptake in the first 30 minute of the experiment was over and over time, the amount of absorption dropped. With increasing absorbent dose, the absorption rate increased, because absorbent dose increase resulted in increased surface area activates and increases the number of active sites absorbed. But in the case of adsorption of phosphate, the change in absorbent dose from 1 g/l to 4 g/l was virtually unaffected. Increased mixing speed is also due to better distribution of nitrate and phosphate molecules in the solution and their contact with adsorbent increased the absorption rate. But in the case of COD measurements, Increase the mixing speed exactly on the contrary, it was absorbed. This means that with increasing mixing speed, the efficiency of COD removal decreased.

According to the results, it is better to remove the COD by the LECA granular is used.

The results of this study are perfectly in line with the results of Asantewah et al (2012) studies on the optimal call time (20 hours). But because of the non-compliance with the linear isotherm model, it is not consistent with the Sharifinia et al (2012), Zarabi et al (2011), Latosinska & Zygadlo (2009) and Azari et al (2014) studies.

The reasons for not matching the results with the Langmuir isotherm model are:

- The fine grains of the absorbent;

- Nonlinearity of the absorption model.

It can be said that this type of absorption follows a nonlinear model.

Using Lightweight Expanded Clay Aggregate (LECA) as a natural and inexpensive absorbent can be used to remove a lot of contaminants.

Since the output of this type of waste as more water will be needed for agriculture and irrigation, therefore, in Table 9, the results obtained from the effluent were compared with national standard and the USEPA¹ (2015) standard. The results indicate that a significant reduction in the amount of COD in both cases (granules and powders) observed which reflects the ability of LECA to absorb pollutants. Nevertheless, the results are still away with national and international standards and the needs for additional process are adsorption.

Table 9. Comparison of the results obtained from the use of LECA with internal and external standards

Factor	Iranian			
	Environmental Protection Organization standards (IRAN DOE) (mg/l)	EPA standard (mg/l) (25)	wastewater passed through LECA (granules) (mg/l)	Wastewater passed through LECA (powder) (mg/l)
BOD ₅	100	30	385	440
COD	200	120	750	850
TSS	100	5	225	260

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