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REMOVAL OF PHOSPHORUS FROM WASTEWATER BY ADSORPTION ONTO NATURAL IRAQI MATERIALS

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ABSTRACT

This study focused on treating wastewater to remove phosphorus by adsorption onto natural and local materials. Burned kaolin, porcelinite, bauxite and limestone were selected to be tested as adsorption materials.The adsorption isotherms were evaluated by batch experiments, studying the effects of pH, temperature and initial phosphorus concentration. The results showed that at pH 6, temperature 20°C and 300 mg/l initial phosphorus concentration; the sorption capacity was 0.61, 9, 10 and 13 mg/g at 10 h contact time, for burned kaolin, porcelanite, limestone and bauxite respectively. As the pH increased from 2 to 10 the removal efficiency for the materials differs in behaviour. The removal efficiency increased from 40 to 90 % for limestone, and decreased from 60 to 30 % for porcelinite. As for bauxite it increased from 60 to 90 % reaching pH 6 then decreased to 30%. Burned kaolin showed the lowest adsorption capacities in these tests. The adsorption isotherms showed that the Langmuir–Freundlich model significantly correlated the experimental data for porcelinite and bauxite, whereby the Freundlich model was best for limestone. The Freundlich and Langmuir–Freundlich models both fit for Burned kaolin. The results show that it is possible to adsorb phosphate from wastewater onto natural Iraqi material and their ability could be ranged as limestone> bauxite> porcelanite> burned kaolin.

KEYWORDS: adsorption, wastewater, phosphorus, bauxite, limestone, porcelanite

الخلاصة

في هذا البحث تمت دراسة طريقة واطئة الكلفة لإزالة الفسفور بالامتزاز وباستعمال مواد طبيعية و محلية. تم اختيار الكاؤولين المحروق، البورسيلينات، البوكسايت و الحجر الكلسي (اللايمستون) كمواد محلية في عملية الامتزاز. بدراسة تأثير المتغيرات من الدالة الحامضية، درجة الحرارة، كمية المادة الممتزة، تركيز الفسفور ومدة التماس على عملية الامتزاز. أظهرت النتائج المختبرية، عند الدالة الحامضية 6، درجة الحرارة 20 درجة مئوية و300 ملغم/ لتر تركيز الفسفور في مياه الفضلات، كانت 0.61، 9، 10و 13 ملغم/غرام بعد مدة تماس 10 ساعات الكاؤولين للبورسيلينات، الحجر الكلسي و .الكمية الممتزة من الفسفور البوكسايت على التوالي. التجربة وعند زيادة الدالة الحامضية من 2 الى 10 تغيرت نسبة إزالة الفسفور لكل مادة بأسلوب متغاير. زادت نسبة الإزالة من 40 الى 90 %، للحجر الكلسي وانخفضت من 60 الى 30 % للبورسيلينات.اما للبوكسايت زادت من 60 الى 90 % عند الدالة الحامضية 6 ثم انخفضت الى 30 %. مادة الكاؤولين المحروق كانت اضعف المواد لامتزاز الفسفور في هذه التجارب. حللت النتائج المختبرية باستخدام موديلات :

Freundlich and Langmuir–Freundlich

اظهرت النتائج يمكن استخدام هذه المواد في امتزاز الفسفور و كفاءة هذه المواد تترتب بالشكل التالي: الحجر الكلسي > البوكسايت > البورسيلينات > الكاؤولين المحروق.

INTRODUTION

Discharge of phosphate loaded wastewater is one of the major sources of phosphorus in water bodies, causing environmental problems such as eutrophication and algae bloom. Different types of natural materials have been tested as adsorption media such as minerals, rocks, soils and marine sediments. These materials can be used as substrates with or without pre-treatment in order to optimize the phosphorus sorption capacity. Among the minerals and rock materials are Dolomite and Dolomite sands, Limestone, Opoka, Wollastonite, Bauxites, Zeolites, and soils such as Marl and Spodosols. Marine sediments such as shell sand and maerl have also been investigated (Del et al., 2003; Siddhesh and Robert, 2004; Bernd and Alan, 2006; Benjamin, 2007). A short description of some of these materials is outlined :

Limestone: is a sedimentary rock that has attracted attention as a candidate substrate for phosphorus sorption due to its high content of calcium mainly $CaCO_3$. The material has been tested in laboratory as well as in field studies. The main mechanisms for phosphorus sorption were the formation of Ca-complexes and precipitation mechanisms (Johansson, 1999).

Opoka: is marine sediment belonging to a group of silica-calcite sediments, found in the deep strata in the eastern part of Europe it has been suggested suitable for wastewater treatment (Johansson, and Gustafsson, 1998; Brogowski and Renman, 2004).

Wollastonite: is a calcium metasilicate mineral, which is a by-product from the mining of Wollastonite and garnet (ferrous metasilicate). The material has been investigated with regard to phosphorus sorption capacity by several researchers (Brooks et al., 2000; Hedstrom, 2006). **Natural Zeolite:** a group of aluminosilicates and comprise a large number of minerals. Zeolites are characterised by high contents of Si- and Al-oxides, and a three-dimensional structure with channels and pores. Different zeolites have been investigated, and the sorption capacities have been calculated according to the Langmuir equation. When mixing zeolites with natural resources for phosphorus adsorption, they demonstrated to have low sorption capacity which results that it is not recommending for phosphorus removal (Sakadevan and Bavor, 1998; Siddhesh and Robert, 2004; Benjamin, 2007).

ADSORPTION MEDIA

In this work four types of natural local materials supplied by the state company of Geological Survey and Mining were used as adsorption medias for phosphate ions. These materials are Bauxite, Porcelanite, Burned kaolin and limestone, which are obtained from various sites at the western part of Iraq. They are taken from industrial beds in

Tarfawi site the Jandily Vally in Al-Rutba region and the west side of Al-Najaf city.The selected materials have been chosen in this work due to their physical and chemical properties which give their ability to absorb phosphorus on their molecular species from the bulk liquid. Also they are of low cost and are locally available.

The chemical analysis of the adsorption media was achieved by using Atomic Absorption Spectrophotometer type PYE UNICAM SP9 in the state company of Geological Survey and Mining. The composition of each material used in this study is presented in Table 1.

The physical properties of the different adsorption media were analyzed in the Oil Research and Development Center-Ministry of Oil. Table 2 represents the physical properties for the selected materials.

ISOTHERM EXPERIMENTS

Batch experiments were adopted to obtain the equilibrium data. Artificial wastewater was prepared from dissolving KH_2PO_4 at different concentrations, 50, 100, 200 and 300 mg/l. Each adsorbent, ranging in weight 0.1, 1, 5 and 10 g were placed in individual 500 ml plastic bottles. The bottles were placed at fixed positions in a water bath with a shaker (Type: Model BS-11). The bottles were left for 15min to 120 hrs to permit adsorption to reach equilibrium conditions. The experiments were conducted at 15, 20, 30 and 40°C and the shaker was fixed at a speed of 100 rpm. The experiments were conducted under controlled and uncontrolled pH conditions. Buffer solutions, 0.1M of H_2SO_4 and NaOH were added drop wise to the water to control the pH. In these runs the pH was controlled at 2, 4, 6, 8 and 10. Buffer solutions were not added to the uncontrolled pH experiments. The details of the experimental runs are illustrated in Tables 3 and 4.

SAMPLES ANALYSIS

A spectrophotometer manufactured by Shimadzu Cooperation model UV-160V was used to identify the absorbance or transmittance of each sample at a wavelength of 690 nm.Each sample was filtered with Whatman filter and diluted to appropriate volume using deionized water. One ml of ammonium molybdate solution and 2 drops of stannous chloride were added to the diluted sample . The solution was mixed and the color was allowed to develop for 10 minutes. The resulting blue colored complex was analyzed by visible range colorimetry method on the spectrophotometer and the concentrations of phosphorus remaining in the solutions were found from the prepared calibration curve

RESULTS AND DISSCUSION

Effect of Initial Phosphate Concentration

Experiments where undertaken to study the effect of various initial concentrations 50, 100, 200 and 300 mg/l at 20°C on phosphate removal using burned kaolin, porcelanite, bauxite and limestone as adsorbents. The removal efficiency of phosphate from the solution and the sorption capacity (q), were calculated. Figures 1-4 indicate a rapid initial uptake rate of phosphate at the beginning and, thereafter, the adsorption capacity remaining almost constant. The adsorption of phosphate reached equilibrium at variable times according to the initial concentration of phosphate and also to the type of the adsorption media. The higher the initial concentration of phosphate species, the larger is the amount of phosphate species adsorbed. The variation in the extent of adsorption increase is due to the fact that initially, all sites on the surface of sorbent were vacant and the solute concentration gradient was relatively high. Consequently the extent of phosphate species uptake decreases significantly with the increase of contact time, which is dependent on the decrease in the number of vacant sites on the surface of adsorption media. After lapse of some time, the remaining vacant surface sites are difficult to be occupied due to repulsive forces between the solute molecules on the solid surface and the bulk phase (Mezenner and Bensmaili, 2008; Krishnan and Haridas, 2008; Boujelben et al., 2008, Zhu et al., 2008).

Figures 1 to 4 show that kaolin has a very low adsorption capacity ranging from 0.1 to 0.6 mg/g when increasing the phosphorus concentration from 50 to 300 mg/L. However, the adsorption capacity was high in bauxite 2-13 mg/g, limestone 2-10 mg/g and in porcelanite 2-9 mg/g, showing good adsorption activity in these materials.

It can be observed from Figures 5 to 8 that the percentage of phosphate removal decreased with increasing the initial phosphate concentration. Very low removal was observed using kaolin reaching 6% at 50 and 100 mg/l phosphate and decreased to 4% for 300 mg/l phosphate. High percentage removal ranging from 80-90% was observed with bauxite for the different concentrations of phosphate. As for limestone, 70 to 80% removal was achieved and 50 to 70% for poreclanite.

The lower uptake at higher concentration resulted from an increased ratio of initial number of moles of phosphate to the available surface area hence fractional adsorption becomes dependent on the initial concentration. For a given adsorption dose the total number of available adsorption sites is fixed thereby adsorbing almost the same amount of adsorbate, thus resulting in a decrease in the percentage removal. Similar conclusions were also observed for the adsorption of phosphate from aqueous solutions onto different materials by Hameed et al., 2007 and Krishnan and Haridas 2008.

EFFECT OF TEMPERATURE

The effect of temperature on the phosphate adsorption rate (q_e) was studied over the temperature range of 15, 20, 30 and 40°C. It can be observed from figures 9-12 that the phosphate adsorption rate on these adsorbents increases with increasing the temperature. This indicates that the adsorption of phosphate ions was favored at higher temperatures. The sorption of phosphate is endothermic, thus the extent of adsorption increased with increasing temperature. Also the difference in the adsorption rate of the studied adsorbents is due to the difference in physical and chemical composition. The maximum adsorption rate was very low in kaolin and increased from about 0.005 to 0.01 mmol/g with increasing the temperature. Bauxite produced the highest adsorption rate 0.14 to 0.15 mmol/g where the rates of porcelanite and limestone were about the same. Similar trend was also observed for phosphate removal reported by Huang et al., 2008; Krishnan and Haridas 2008; Boujelben et al., 2008 and Zhu et al., 2008.

EFFECT OF PH

To examine the effect of pH on the adsorption of phosphate, batch experiments using phosphate solution with initial concentration of 300 mg/l were conducted. Ten grams of each adsorbent was added to 500 ml of phosphate solution. The pH of the solution increased from 2, 4, 6, 8 and 10. Figure 13 show the effect of pH on the removal efficiency for the selected medias at 10 h contact time and 20^0C. A very slight effect of pH on the performance of burned kaolin can be observed where the removal efficiency didn’t exceed 4%. For porcelanite and bauxite adsorbents, the removal efficiency increased as the pH increased and reached a maximum value of 65% at pH 4 for porcelanite and 85% at pH 6 for bauxite. As the pH increased to 10, the removal efficiency decreased gradually to about 35% for the two medias. For limestone the situation was different, as the pH increased the removal efficiency increased from 35% to 90%.

MODELING OF SORPTION EQUILIBRIUM

In order to optimize the design of an adsorption system to remove phosphate from aqueous solutions, it is important to establish the most appropriate correlation for the equilibrium curves. The isotherm data were analyzed using three of the mostly used equilibrium models. Langmuir, Freundlich and Langmuir-Freundlich.

The experiments were conducted at temperatures 15, 20, 30 and 40°C for 10 g adsorbent dose, pH 6, and the initial phosphate concentration ranging 50, 100, 200 and 300 mg/l. The Langmuir model is an ideal model for homogenous monolayer adsorption while the langmuir-Freundlich model is obtained by introducing a power law expression of the Freundlich model into the Langmuir model. The best fit values of the model parameters estimated by linear regression analyses are listed in Tables 5-8 for the different materials. The correlation coefficients (R^2), provides measurement of model fitness. A comparison of the experimental isotherms with the adsorption isotherm models showed that the Langmuir equation represented the poorest fit of the experimental data for burned kaolin as compared to the other isotherm equations. For poreclanite, bauxite and limestone the best fits are Freundlich, Langmuir-Freundlich and Freundlich, repetitively.

CONCLUSIONS

The results of the present study can provide a process for developing a low-cost technology based on adsorption by burned kaolin, porcelanite, bauxite and limestone for phosphate removal from wastewater. The following points can be concluded from the present study:

- Burned kaolin, porcelanite, bauxite and limestone could be employed as adsorbents in the removal of phosphates and their ability could be arranged in the following order: Limestone> Bauxite> Porcelanite> Burned kaolin
- As the initial concentration of phosphate species increases 50 to 300 mg/l, the amount of adsorbed phosphate species on the adsorbent increases. The adsorption capacity in bauxite and limestone and porcelanite ranged 2-13 mg/g with increasing the phosphate concentration in the solution.
- The percent of phosphate removal decreases as the initial concentration of phosphate increases 50 to 300 mg/l. It increased 80-90% in bauxite, 70-80% in limestone and 50-70% in poreclanite. Kaolin showed very low removal efficiency.
- As the temperature increases 15-40°C the adsorption rate increases over the studied adsorbent which indicates that

the adsorption of phosphate was favoured at higher temperature, since the adsorption of phosphate is endothermic.Bauxite showed the highest rate 0.14-0.15 mmol/g where porcelanite and limestone were about the same. Very low rates 0.005-0.01 mmol/g was observed in kaolin.

- The adsorption process is highly dependent on the pH of the solution. For porcelanite and bauxite the removal efficiency increased as the pH increased and reached a maximum value of 65% at pH 4 and 85% at pH 6 for porcelanite and bauxite respectively. Then the removal efficiency decreased. For limestone the situation was different, the removal efficiency increased and reached a maximum value 90% at pH 10.
- The adsorption isotherms of phosphate could be well described by Langmuir-Freundlich models for porcelanite and bauxite, while the Freundlich model for limestone. The two models, Freundlich and Langmuir-Freundlich, could be used for burned kaolin.

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Table 1 Chemical Composition of Adsorption Media

Material	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	SO_3	L.O.I
	Wt.%						
Bauxite	30.25	60.59	1.19	2.48	6.45	-	0.14
Porcelanite	59.5	28.25	1.08	<1	<1	-	0.1
Burned Kaolin	53.95	40.49	1.21	0.95	2.46	0.80	0.14
Limestone	5.000	4.000	2.50	2.50	85.3	-	0.7

Table 2 Physical Properties of the Adsorption Media

Material	Surface Area, m^2/g	Pore Volume cm^3/g	Bulk Density g/cm^3
Bauxite	82.02	0.18	0.78
Porcelanite	16.68	0.25	1.05
Burned Kaolin	0.23	0.03	1.10
Limestone	9.17	0.26	0.83

Table 3 Controlled pH Experiments

Run	Temp. ˚C	pH	Time hr	Phosphate concentration mg/l	Weight of adsorbent g	Particle mesh no.	Type of adsorbent
1	20	2, 4, 6, 8 and 10	0.25 -120	300	10	(8-16)	Limestone
2	20	2, 4, 6, 8 and 10	0.25 -120	300	10	(8-16)	Kaolin
3	20	2, 4, 6, 8 and 10	0.25 -120	300	10	(8-16)	Bauxite
4	20	2, 4, 6, 8 and 10	0.25 -120	300	10	(8-16)	Porcelanite

Table 4 Uncontrolled pH Experiments

Run	Temp., ˚C	Time hr	Initial concentration mg/l	Weight of adsorbent g	Particle mesh no.	Type of adsorbent
1	15, 20, 30 and 40	0.25 up to 48	50, 100, 200 and 300	0.1, 1, 5 and 10	(8-16)	Limestone
2	15, 20, 30 and 40	0.25 up to 48	50, 100, 200 and 300	0.1, 1, 5 and 10	(8-16)	Kaolin
3	15, 20, 30 and 40	0.25 up to 48	50, 100, 200 and 300	0.1, 1, 5 and 10	(8-16)	Bauxite
4	15, 20, 30 and 40	0.25 up to 48	50, 100, 200 and 300	0.1, 1, 5 and 10	(8-16)	Porcelanite

Table 5 Isotherm Constants for Phosphate Adsorption onto Burned kaolin

Parameters	**Temperature, °C**			
	15	**20**	**30**	**40**
Langmuir				
q_m mmol/g	**0.011**	**0.011**	**0.012**	**0.015**
b (L/mmol)	**0.327**	**0.482**	**0.594**	**0.709**
R^2	**0.974**	**0.981**	**0.966**	**0.993**
Freundlich				
n	**1.073**	**1.584**	**1.509**	**1.699**
k_f ($mmolL^{-1/n}g^{-1}L^{1/n}$)	**0.003**	**0.003**	**0.005**	**0.520**
R^2	**0.992**	**0.998**	**0.994**	**0.997**
Langmuir-Freundlich				
n	**0.998**	**0.991**	**0.975**	**1.010**
k (mmol/L)	**2.725**	**2.744**	**2.780**	**26.60**
R^2	**0.994**	**0.993**	**0.978**	**0.994**

Table 6 Isotherm Constants for Phosphate Adsorption onto Porcelanite

Parameters	**Temperature, °C**			
	15	**20**	**30**	**40**
Langmuir				
q_m mmol/g	**0.138**	**0.173**	**0.176**	**0.156**
b (L/mmol)	**0.851**	**0.736**	**1.389**	**3.433**
R^2	**0.985**	**0.966**	**0.975**	**0.983**
Freundlich				
n	**1.465**	**1.361**	**1.452**	**1.780**
k_f ($mmolL^{-1/n}g^{-1}L^{1/n}$)	**0.061**	**0.073**	**0.109**	**0.139**
R^2	**0.989**	**0.986**	**0.965**	**0.952**
Langmuir-Freundlich				
n	**0.998**	**1.019**	**1.047**	**1.018**
k (mmol/L)	**2.722**	**2.670**	**130.1**	**122.8**
R^2	**0.989**	**0.987**	**0.968**	**0.955**

Table 7 Isotherm Constants for Phosphate Adsorption onto Bauxite

Parameters	**Temperature, °C**			
	15	**20**	**30**	**40**
Langmuir				
q_m (mmol/g)	**0.960**	**0.260**	**0.227**	**0.205**
b (L/mmol)	**0.332**	**4.816**	**3.447**	**18.88**
R^2	**0.953**	**0.961**	**0.999**	**0.951**
Freundlich				
n	**1.073**	**1.403**	**1.509**	**1.699**
k_f ($mmolL^{-1/n}g^{-1}L^{1/n}$)	**0.264**	**0.404**	**0.248**	**0.520**
R^2	**0.999**	**0.998**	**0.991**	**0.878**
Langmuir-Freundlich				
n	**1.003**	**0.999**	**1.039**	**0.987**
k (mmol/L)	**283.7**	**19.82**	**25.00**	**4.953**
R^2	**0.999**	**0.999**	**0.992**	**0.954**

Table 8 Isotherm Constants for Phosphate Adsorption onto Limestone

Parameters	**Temperature, °C**			
	15	**20**	**30**	**40**
Langmuir				
q_m mmol/g	**0.227**	**0.185**	**0.134**	**0.133**
b (L/mmol)	**0.829**	**1.336**	**6.713**	**16.29**
R^2	**0.988**	**0.956**	**0.989**	**0.987**
Freundlich				
n	**1.313**	**1.505**	**2.286**	**3.142**
k_f ($mmolL^{-1/n}g^{-1}L^{1/n}$)	**0.107**	**0.109**	**0.131**	**0.142**
R^2	**0.993**	**0.998**	**0.996**	**0.991**
Langmuir-Freundlich				
n	**0.997**	**1.037**	**1.081**	**1.304**
k (mmol/L)	**115.9**	**62.56**	**11.25**	**3.098**
R^2	**0.998**	**0.999**	**0.973**	**0.902**

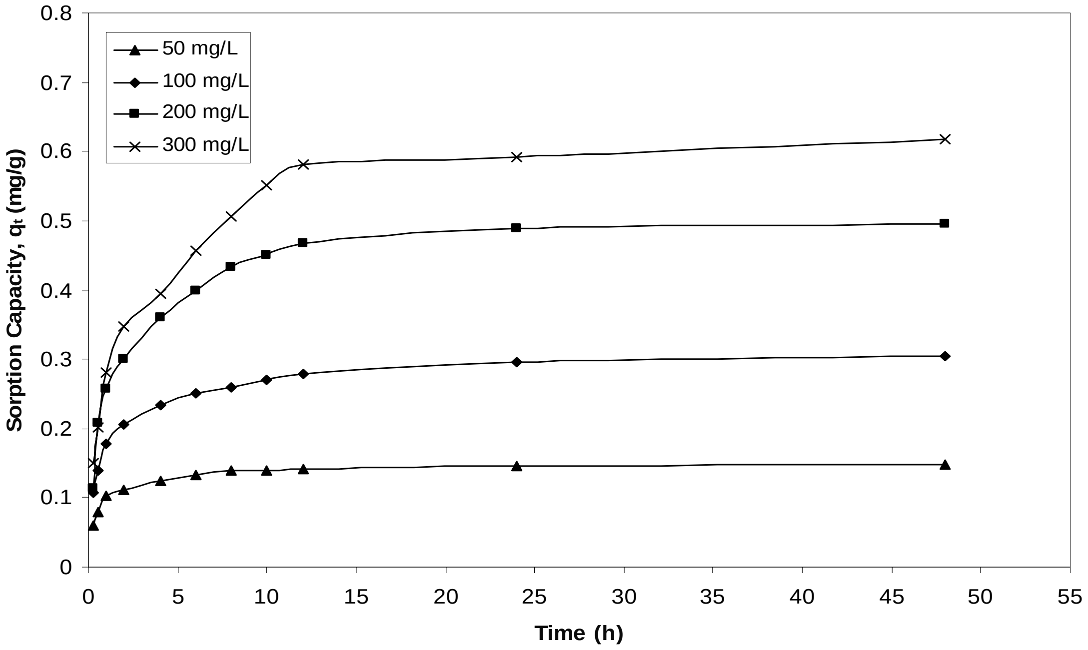


Figure 1 Variation of Sorption Capacity for burned kaolin at several initial phosphate concentrations at 20°Cand pH 6.

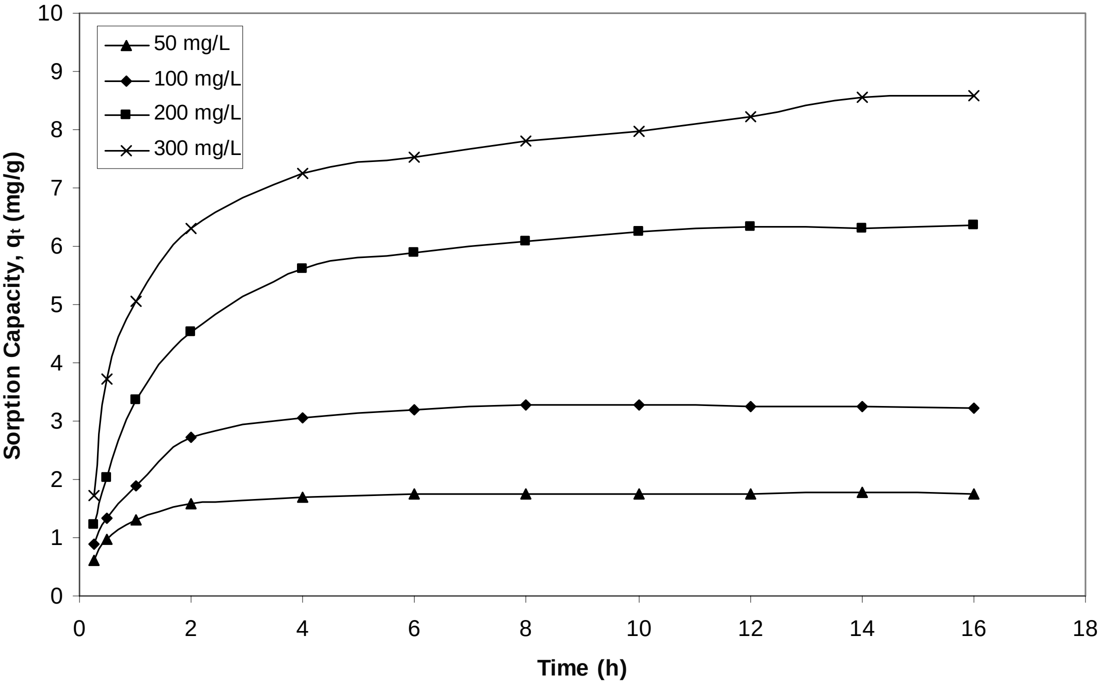


Figure 2 Variation of Sorption Capacity for Porcelanite at several initial phosphate concentrations at 20°Cand pH 6.

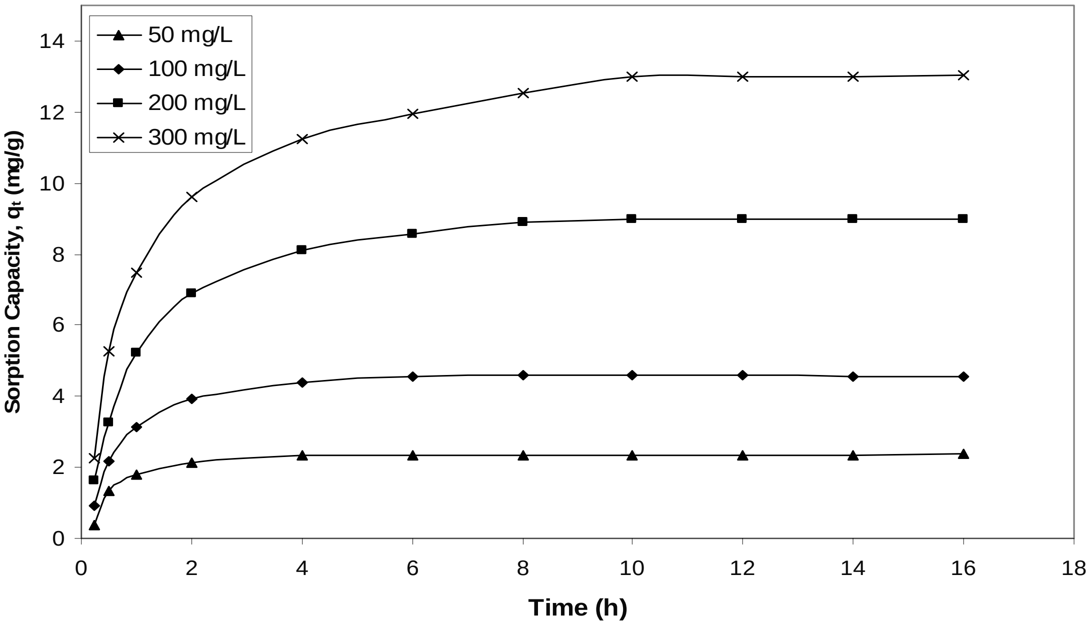


Figure 3 Variation of Sorption Capacity for Bauxite at several initial phosphate concentrations at 20°Cand pH 6.

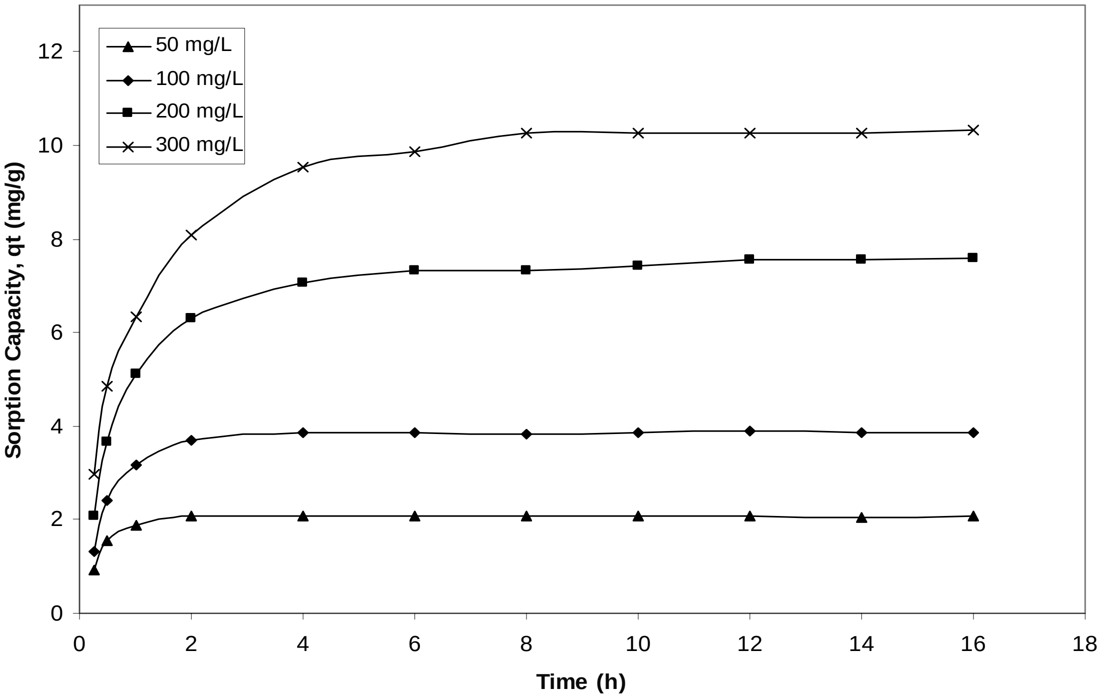
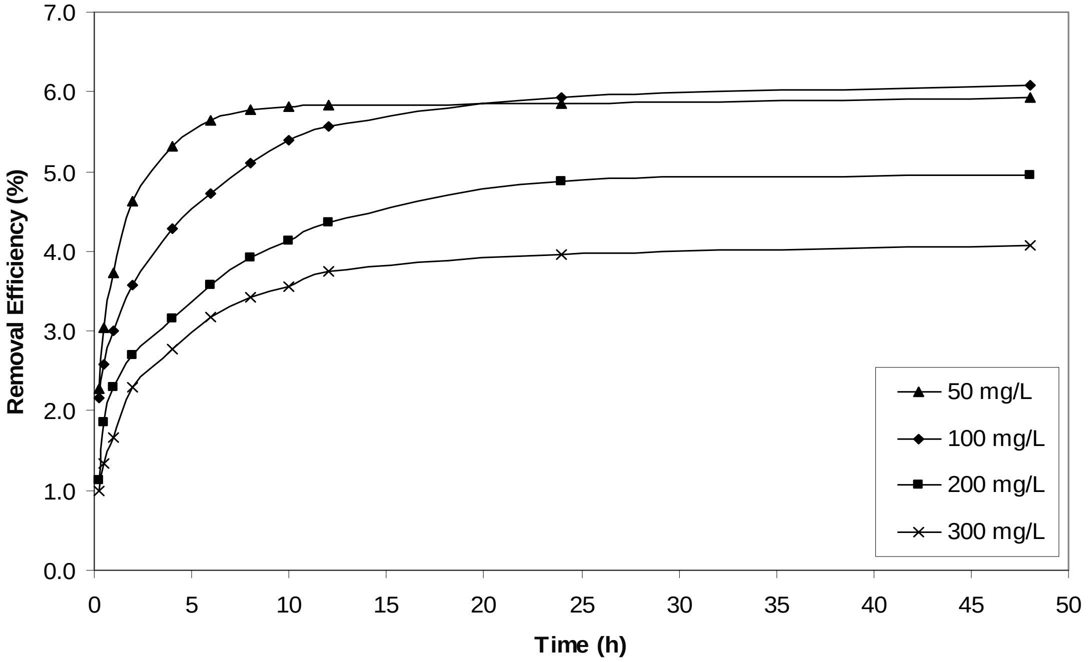


Figure 4 Variation of Sorption Capacity for Lmestone at several initial phosphate concentrations at 20°Cand pH 6.



Figurer 5 Effect of initial concentration of phosphate ions in the solution on removal efficiency onto Burned kaolin at 20°C, pH 6.

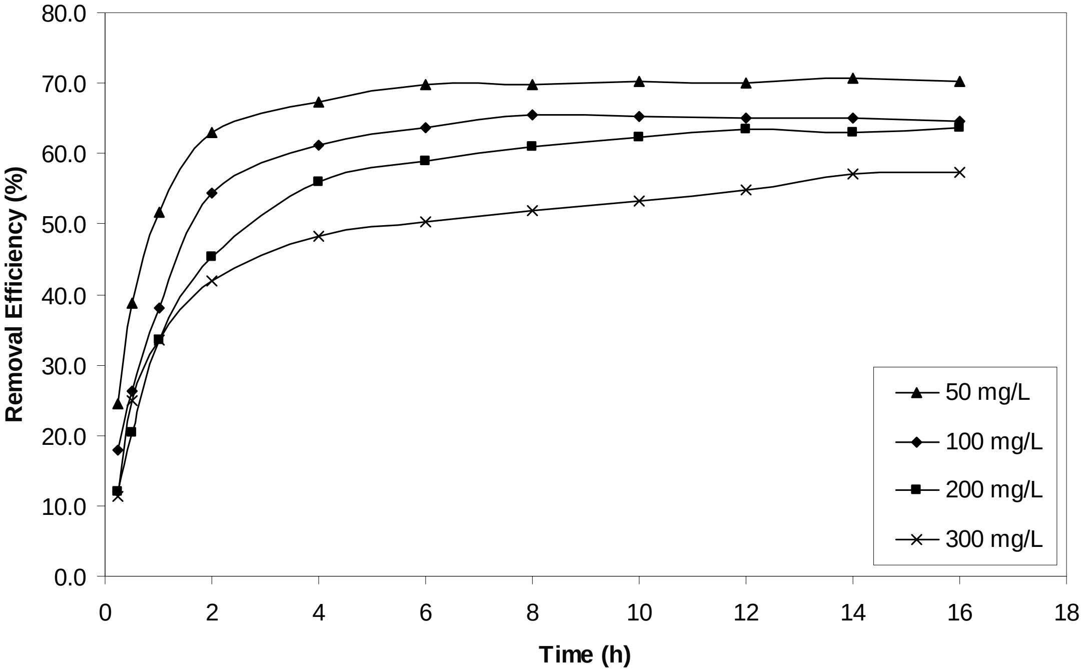


Figure 6 Effect of initial concentration of phosphate ions in the solution on removal efficiency onto Porcelanite at 20°C, pH 6.

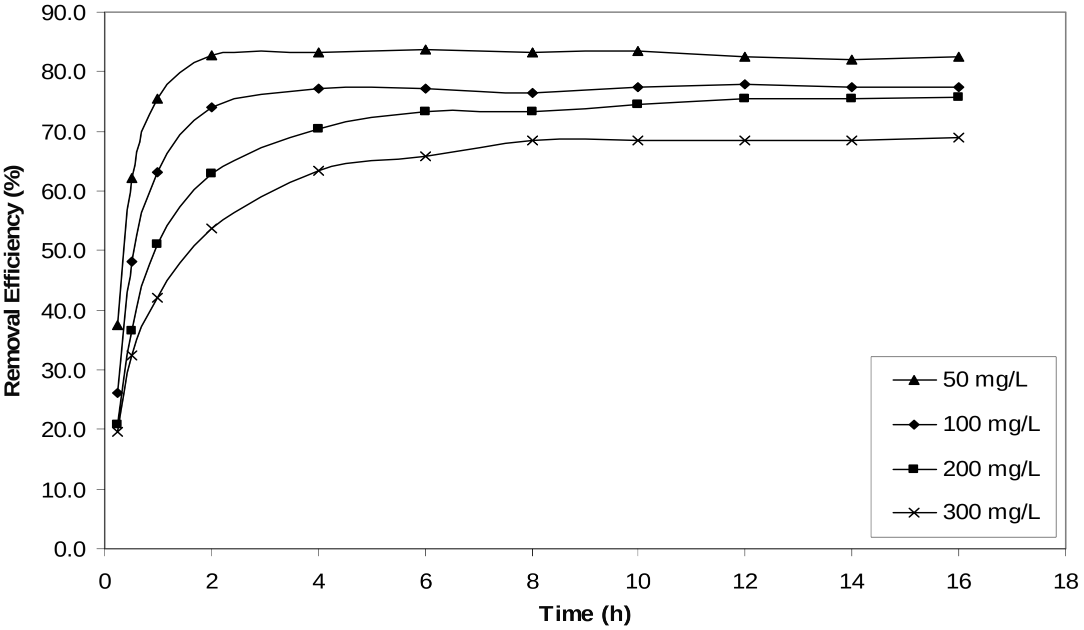


Figure 7 Effect of initial concentration of phosphate ions in the solution on removal efficiency onto Bauxite at 20°C, pH 6.

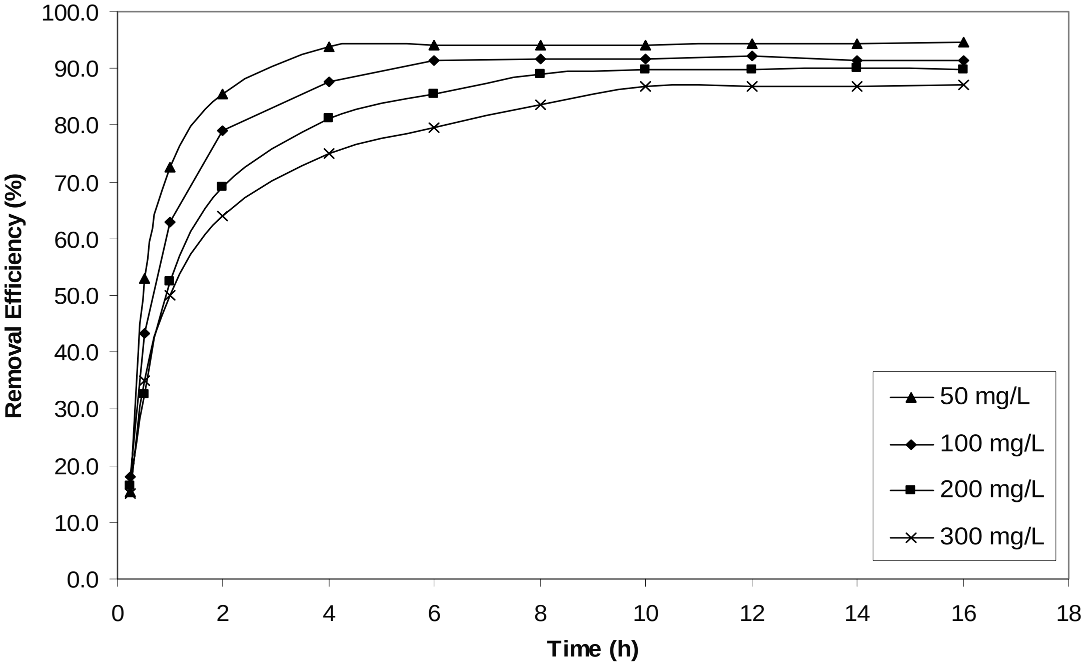


Figure 8 Effect of initial concentration of phosphate ions in the solution on removal efficiency onto Limestone at 20°C, pH 6.

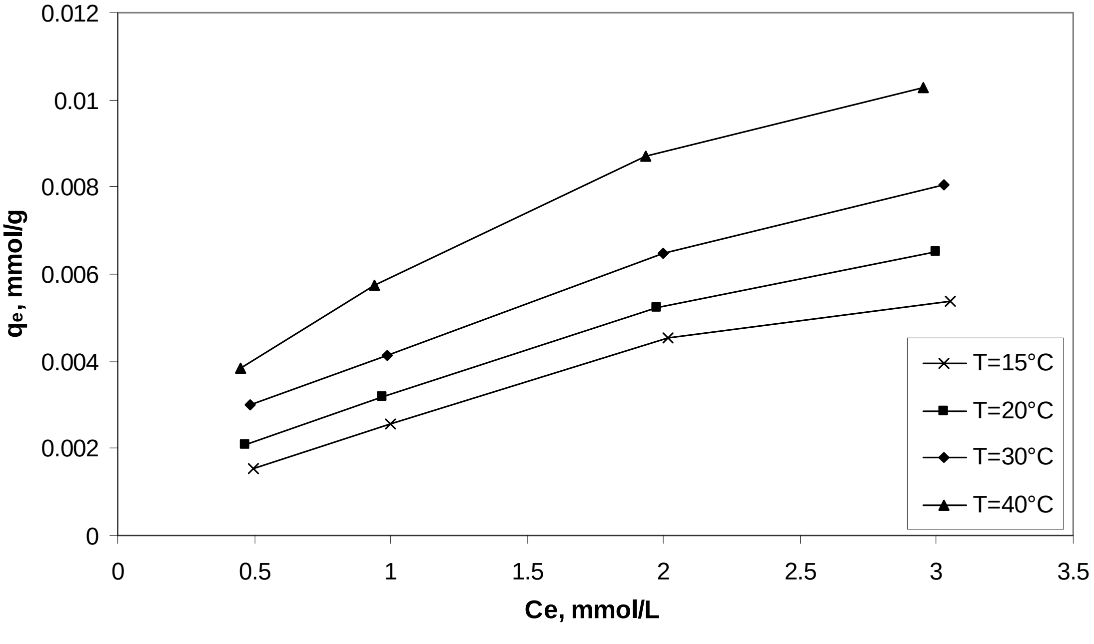


Figure 9 Phosphate adsorption rate at various temperatures using Burned kaolin as adsorbent. (Contact time 10h, dose 10g and pH 6).

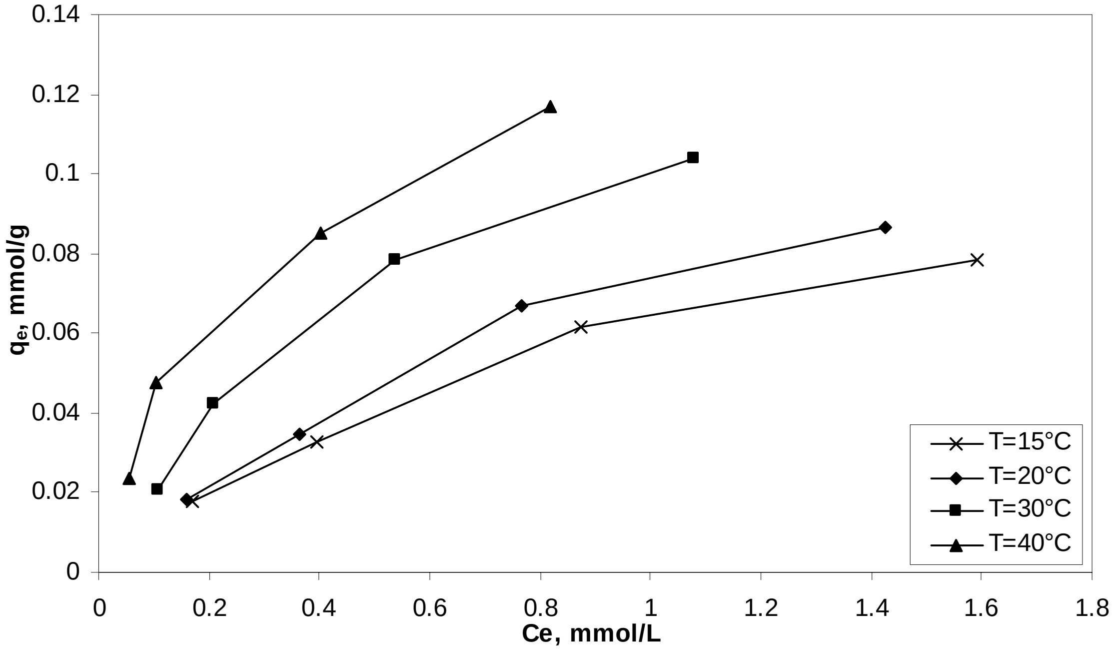


Figure 10 Phosphate adsorption rate at various temperatures using Porcelanite as adsorbent. (Contact time 10h, dose 10g and pH 6).

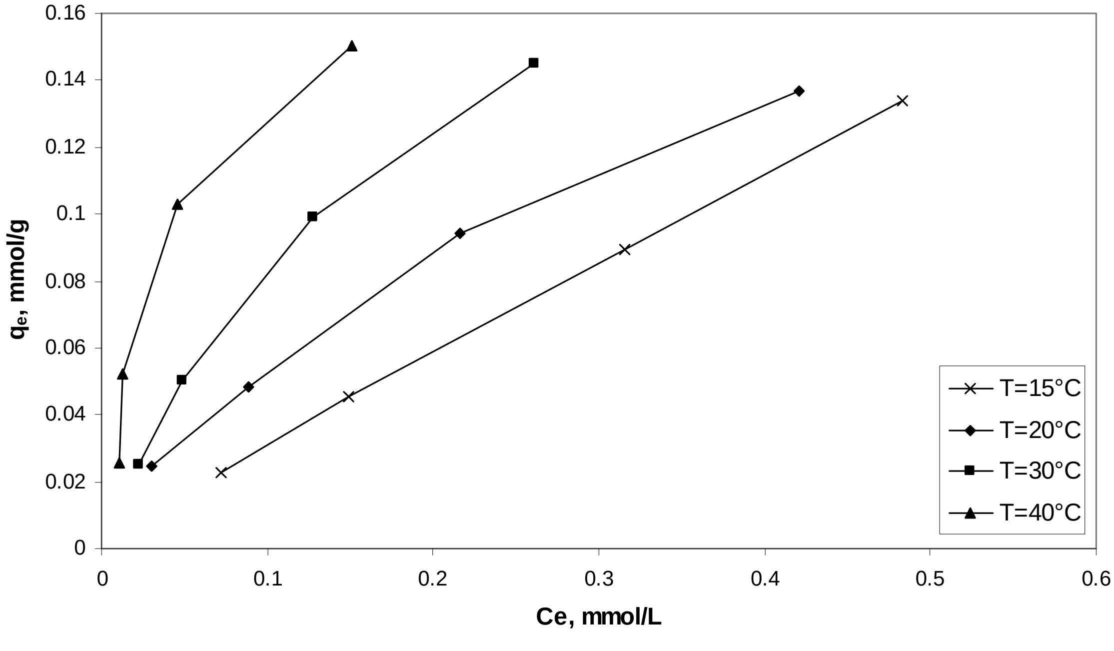


Figure 11 Phosphate adsorption rate at various temperatures using Bauxite as adsorbent. (Contact time 10h, dose 10g and pH 6).

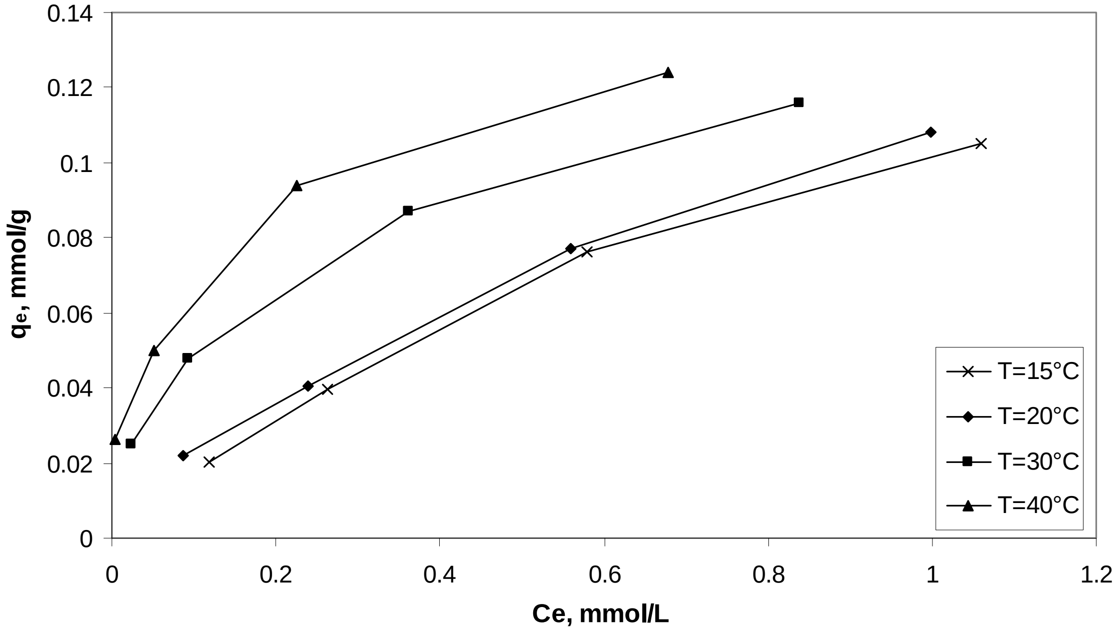


Figure 12 Phosphate adsorption rate at various temperatures using Limestone as adsorbent. (Contact time 10h, dose 10g and pH 6).

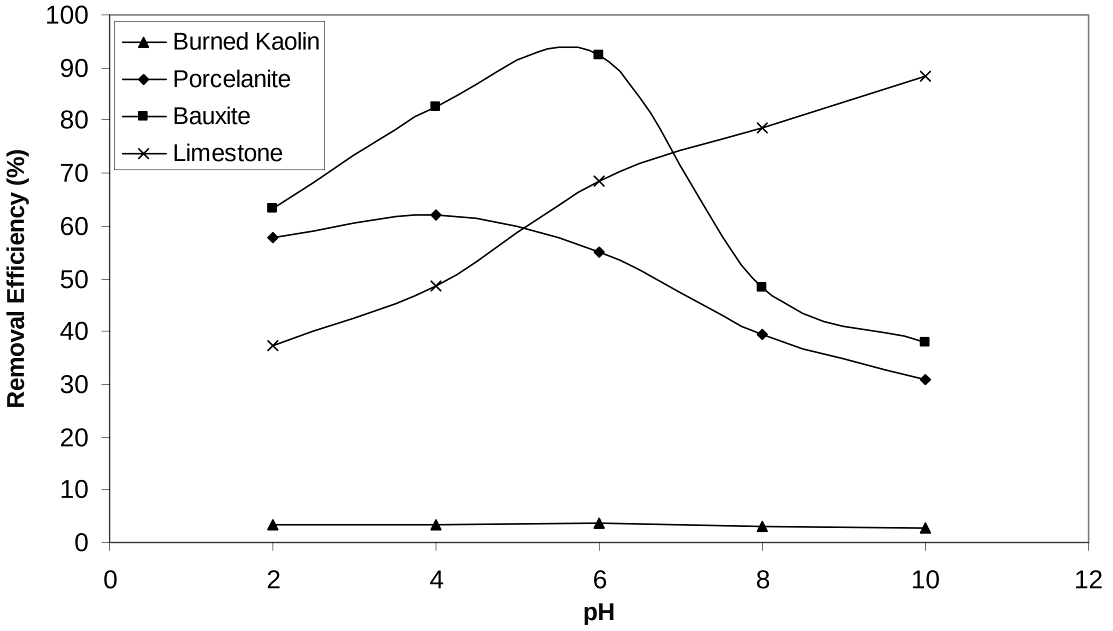


Figure 13 Effect of pH on the adsorption removal efficiency using different adsorption media (initial phosphate concentration 300 mg/l, dose 10g and temperature 20°C and contact time 10 h).