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Research Article

The Use of Synthesized Zinc Oxysulphide Nanoparticles in Phosphate Phosphorus Removal from Synthetic Wastewater and Statistical Analysis

Nilüfer Ülgüdür

Düzce University, Faculty of Engineering, Department of Environmental Engineering, Düzce, Türkiye, niluferulgudur@duzce.edu.tr, ror.org/04175wc52

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ABSTRACT

This study investigated the phosphate phosphorus (PO_4-P) removal potential of zinc oxysulfide (ZnO_xS_y) nanoparticles obtained by fifteen varying component ratios. The statistical meaning of the distinct synthesis compositions was evaluated by regression analysis based on the response of PO_4-P removal efficiencies. The results indicated that ZnO_xS_y nanoparticles could remove PO_4-P by 99.5% without optimization of the adsorption process (Initial PO_4-P concentration: 15 mg/L, adsorbent dose: 1 g/L, pH: 4.31, contact time: 2 hr). However, the synthesis compositions of ZnO_xS_y nanoparticles strongly effect the PO_4-P removal efficiency. The data could be interpreted by regression analysis with a high R^2 of 89.61% and p value of 0.000. The main component that positively affect the PO_4-P removal efficiency was hydrogen peroxide, whereas sodium sulfide component had a limited effect.

1. Introduction

Phosphorus is a nutrient for living things. However, it can be regarded as a pollutant when severe consequences of its prevalence in water bodies such as eutrophication is considered. Therefore, phosphorus pollution in water bodies should be controlled by eliminating the discharges of or treating phosphorus containing wastes and wastewaters [1, 2]. The oxidation states of phosphorus are +II, +III, +IV, and +V and the coordination numbers range from one to six [3]. The removal of phosphorus from water should have supposedly been simple depending on the positive charge related to the oxidation state of the phosphorus. Since the positively charged pollutants can be removed by conventional adsorbents such as zeolites and clay due to their opposite (negative) surface charges [4].

However, phosphorus forms oxyanions in water (i.e. dihydrogen phosphate- $H_2PO_4^-$, hydrogen phosphate -HPO_4^{2-} and phosphate- PO_4^{3-}), which can convert positively charged phosphorus element into negatively charged compounds by attracting oxygen elements in water depending on the pH value [5]. Thus, the removal of phosphorus from aqueous solutions become challenging.

Several methods have been offered for phosphorus removal from aqueous environment such as physico-chemical, biological and/or combinations [6, 7]. However, these methods have several limitations that can be regarded as the lack of cost-effectiveness and high removal efficiencies, the generation of toxic products or wastes which requires to be further handling [7]. Adsorption processes have been considered as a widely applicable pollutant removal process based on the high removal efficiencies, cost-effectiveness and easy application [8-10]. Nanoparticles and the derivative composites are emerging adsorbents for phosphorus removal due to their reactivities, selectivity and high surface areas [11,12].

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Lanthanum-zirconium binary metal oxide nanoparticles [10] and ceria-loaded biochar [12] are among the emerging adsorbents produced with the aim of phosphate phosphorus (PO_4-P) removal. These adsorbents offer high removal efficiencies, selectivity for PO_4-P and fast removal kinetics [11, 12]. However, the use of rare earth metals such as lanthanum and ceria, can increase the overall cost of the process due to their lower abundance and relatively high costs [13].

Relatively economical alternatives of the nanoparticles can also be used as PO_4-P adsorbents. Nanoscale zero valent iron (nZVI) has been widely used adsorbents in water treatment due to its reaction and reduction potential with most of the pollutants in aqueous environment. nZVI dose of 1 g/L can remove 76.8 mg PO_4-P when the initial concentration is 100 mg/L [14], while silica nanoparticles obtained from rice husk ash had PO_4 adsorption capacity of 9.08 mg/g [15]. Iron-manganese oxide spinel with $MnFe_2O_4$ structure exhibited PO_4-P adsorption efficiency of 98.52% (adsorbent dosage: 2.5 g/L, PO_4-P concentration: 10 ppm) [16].

These studies apparently demonstrate the efficiency of nanoparticles in PO_4-P removal from aqueous environment. However, the variability in synthesis conditions of nanoparticles can considerably affect the key features of the adsorbents such as particle size, shape and functional groups [17-19]. Therefore, the synthesis conditions should be optimized depending on the target pollutant to identify the critical components in the synthesis to reach an efficient treatment process.

Zinc (Zn) is one of the abundant, low-cost and environmentally friendly material [20], which can be used in adsorbent synthesis. Furthermore, Zn can improve the selectivity in adsorption processes for enhancing PO_4-P removal [21, 22]. To date, Zn has been incorporated into nanoparticles and their composites such as in the form of CaZnFeZr; MgFeZr and MgZnFe [21]. On the other hand, zinc oxysulfide (ZnO_xS_y) nanoparticles have been synthesized and used for the removal of another oxyanion, i.e. arsenic, from aqueous solutions [23]. However, there is no study investigated PO_4-P removal using ZnO_xS_y nanoparticles.

This study aims at identification of the optimum chemical synthesis conditions of ZnO_xS_y nanoparticles for the removal of PO_4-P from aqueous environment. To this purpose, ZnO_xS_y nanoparticles were synthesized under varying compositions of the chemical components. Batch PO_4-P adsorption tests were performed for each synthesis. The results were evaluated by fitting a regression model to reach an efficient PO_4-P adsorbent.

1. General Methods

1.1. Synthesis of zinc oxysulfide particles

ZnO_xS_y particles were synthesized according to the method proposed by Uppal et al [23]. Briefly, zinc chloride ($ZnCl_2$) was dissolved in deionized water. Concentrated ammonium hydroxide solution (NH_4OH, 25%, will be denoted as NH_4) was added to the solution. Sodium sulfide nonahydrate ($Na_2S.9H_2O$, will be denoted as Na_2S) and hydrogen peroxide (H_2O_2, 30%) were added to the solution, respectively. The process for the formation of ZnO_xS_y was carried out under continuous stirring and at approximately 90°C. The formed precipitate was filtered and washed thoroughly with deionized water. ZnO_xS_y nanoparticles were then dried in the oven at 65°C overnight. Fifteen forms of ZnO_xS_y nanoparticles with varying components were synthesized and used in the study.

1.2. Batch adsorption tests

Synthetic phosphate solution was prepared by dissolving potassium dihydrogen phosphate (KH_2PO_4) in deionized water. The initial concentration of PO_4-P was set to 15 mg/L. The initial pH of the solution was 4.31. The adsorbent dose was 1 g/L. The batch adsorption experiment was performed for each adsorbent in a rotary mixer at 70 rpm using 50 mL centrifuge tubes. The solution was immediately filtered from 0.22 μm pore-sized nylon syringe filters.

PO_4-P was measured spectrophotometrically by ascorbic acid method according to the Standard methods [24]. The following formula was used to calculate the removal of the adsorption process [25]:

$$R(\%) = \frac{(C_o - C_e)}{C_o} x100 \quad (1)$$

where C_o and C_e represent the initial and final concentrations of PO_4-P in the solution (mg/L) and R represents the PO_4-P removal efficiency.

1.3. Regression model analysis

Minitab Statistical Software 22 was used to identify the optimum regression equation considering the system components. This equation representing the best correlation between the synthesis components and PO_4-P removal efficiency had a significant statistical meaning ($p<0.05$).

3. Results and Discussion

3.1. The synthesis of zinc oxysulfide particles and adsorption tests

The synthesis compositions of components of $ZnCl_2$, NH_4, Na_2S and H_2O_2 are given in Table 1. A white precipitate initially formed as NH_4 was added and then disappeared as more NH_4 was added. NH_4 of 0.5 mL was the initial point for the precipitate formation, whereas 3 mL represented the initial stage for precipitate disappearance. The amount of Na_2S were determined based on the weight ratios of 1:0.5 (Zn-1, Zn-2, Zn-4, Zn-11, Zn-13), 1:1 (Zn-3, Zn-5, Zn-6, Zn-12, Zn-14) and 1:5.25 (Zn-7, Zn-8, Zn-9, Zn-10, Zn-15). H_2O_2 was added in 0, 2 and 10 mL in different synthesis.

The PO_4-P removal efficiencies ranged approximately from 4.0% to 99.5% using fifteen individual syntheses (Table 1). This change indicated that the variances in synthesis conditions had significant effect on the removal efficiency of the target pollutant from aqueous solutions [17]. The adsorption test was conducted to compare the PO_4-P removal efficiency of ZnO_xS_y nanoparticles without any optimization of batch adsorption tests. Indeed, the high removal efficiency using Zn-2 nanoparticles (removal: 99.5%, initial PO_4-P concentration: 15 mg/L, adsorbent dose: 1 g/L, pH: 4.31, contact time: 2 hr) revealed that ZnO_xS_y had significant PO_4-P removal efficiency from aqueous environment. The lowest PO_4-P removal efficiency was observed using Zn-7 nanoparticles under the same operating conditions.

3.2. Evaluation of the regression model

Minitab Statistical Software 22 was used to obtain best applicable regression model. This method is particularly useful to define the relationship between one **dependent parameter** (removal efficiency) and one or more **independent parameter** (predictors) [26]. The independent parameters were determined as NH_4, Na_2S and H_2O_2. The order of interactions and terms were adjusted in model, which enable the model to be flexible enough to capture complex, non-linear, and interactive effects of NH_4, Na_2S and H_2O_2 on removal efficiency. The two-sided confidence interval (higher and lower) around the estimated value indicated that the true value would fall within this range with 95% probability. The 95% level of confidence is generally acceptable in most scientific and industrial applications. Each parameter was evaluated by Type III sum of squares to show the contribution of each parameter while accounting for all others [27].

Several regression models were obtained until the regression equation had a statistical meaning, which was regarded as a p value below 0.05 [28]. The statistically acceptable regression model equation (with a p value of 0.000) represented the mathematical relationship between the predictors of NH_4, Na_2S and H_2O_2 and the response parameter (PO_4-P removal efficiency) according to analysis of variance (ANOVA) analysis. The regression model is presented in Equation 2.

$$R = 56.1\ NH_4 - 1.05\ Na_2S + 5.34\ H_2O_2 - 20.8\ NH_4^2 + 1.093\ NH_4^3 \quad (2)$$

where NH_4 in mL, Na_2S in g and H_2O_2 in mL, R in %.

Table 1. The components used in the synthesis of ZnO_xS_y nanoparticles and the adsorbent codes

Adsorbent	$ZnCl_2$, g	NH_4, mL	Na_2S, g	H_2O_2, mL	PO_4-P removal, %
Zn-1	3 g	0.50	1.50	10.00	90.024
Zn-2	3 g	2.00	1.50	10.00	99.525
Zn-3	3 g	0.50	3.00	10.00	98.575
Zn-4	3 g	3.00	1.50	10.00	40.380
Zn-5	3 g	2.00	3.00	10.00	80.523
Zn-6	3 g	3.00	3.00	10.00	59.857
Zn-7	3 g	2.00	15.75	0.00	4.038
Zn-8	3 g	3.00	15.75	0.00	19.715
Zn-9	3 g	2.00	15.75	2.00	18.290
Zn-10	3 g	3.00	15.75	2.00	13.777
Zn-11	3 g	16.00	1.50	0.00	65.321
Zn-12	3 g	16.00	3.00	0.00	74.347
Zn-13	3 g	16.00	1.50	2.00	12.352
Zn-14	3 g	16.00	3.00	2.00	68.884
Zn-15	3 g	16.00	15.75	10.00	73.159

The regression model presented three different coefficient of determination (R^2) values as R-sq, R-sq(adj), and R-sq(pred) (Table 2). R-sq value was 89.61% which indicated that the model explained 89.61% of the variability in the removal efficiency based on the amounts of NH_4, Na_2S, and H_2O_2 used. This suggested a strong correlation between the model and the input parameters. R-sq(adj) value (84.42%) took the number of predictors in the model into account, to prevent overfitting. R-sq(adj) value indicated that the model had a correlation of 84.42% among the parameters after adjusting the number of input parameters in the model [29].

The high R-sq(adj) value suggested that the model parameters were effective, and the model is not overfitted by including unnecessary predictors. R-sq(pred) was observed to be 71.44%, which was a value to assess the predictive power of the model for the data that was not included in modeling data set. R-sq(pred) is calculated by a formula that eliminates an observation from the data set, estimates the regression equation, and assesses how well the model predicts the eliminated observation [30]. Even though the predictive power was lower than R-sq, the values of R-sq and R-sq(pred) were compatible.

The normal probability plot of residuals (errors) indicates whether the residuals are approximately normally distributed or not, which represents one of the key assumptions of linear regression on model fitting to the data. The residuals of the data were mostly on or close to the redline (Figure 1a). This fact indicated the residuals were almost normally distributed.

Table 2. The regression model fitting parameters

Regression model	R-sq		89.61%
	R-sq(adj)		84.42%
	R-sq(pred)		71.44%
Terms	Coefficient	T-Value	p-Value
NH_4	56.1	1.97	0.077
Na_2S	-1.05	-0.87	0.402
H_2O_2	5.34	3.57	0.005
NH_4^2	-20.8	-1.94	0.081
NH_4^3	1.093	1.94	0.081

Additionally, the histogram of the residuals (Figure 1b) controls whether the residuals fit to the normal distribution. The residuals are not perfectly resembled a normal distribution as indicated also by the normal probability of the plot (Figure 1a). The most negative residual on the normal probability plot suggested the potential presence of an outlier. However, repeated synthesis and batch adsorption tests confirmed that this data point is a valid observation and not an outlier. This indicated that the observed deviation could stem from the inherent variability of the system. Alternatively, it could result from limitations in the model's ability to fully capture all underlying factors influencing the response. The residuals versus fits plot serves for comprehending how randomly residuals are distributed with respect to fitted values. The residuals versus fits plot (Figure 1c) showed a limited random distribution of residuals. This suggests that the predictive capability of the model can be improved through further refinement, such as additional synthesis experiments or batch adsorption tests to better capture the variability in the system. The random distribution of the residual versus observation order plot (Figure 1d) indicated that there are no time- or order-related biases, and the performance of the model is consistent over the experimental sequences.

3.3. The effect of components used in the synthesis on removal efficiency

Linear NH_4 term had a p-value 0.077. This was slightly higher than the threshold of statistically meaningful data (0.05). It had a large coefficient in the regression model equation (56.1) (Table 2). The p values of the quadratic (NH_4^2) and cubic terms (NH_4^3) of NH_4 (0.081) were also higher than 0.05. These facts suggested that NH_4 could have a significant positive but not a definite impact on the removal efficiency. The positive effect indicates that the larger the amount of NH_4, the more the removal of PO_4-P from the aqueous solution. However, the coefficients of NH_4^2 (-20.8) and NH_4^3 (1.093) revealed that as the volume of NH_4 increased at very high levels, the positive impact on the removal of PO_4-P diminished or even turned to negative.

Na_2S having a p value of 0.402 was statistically insignificant, which reflected Na_2S did not have a considerable effect on the removal efficiency. However, it should be noted that the addition of Na_2S is a compulsory application to form ZnO_xS_y nanoparticles. Therefore, the evaluation of 'statistically insignificant' was based on the range of Na_2S amounts added during the synthesis. Na_2S has a little decreasing effect on removal efficiency, considering the coefficient in the regression equation (-1.05). H_2O_2 component influences the removal efficiency, based on the p value as statistically significant (p: 0.005). The positive coefficient of H_2O_2 in regression analysis (5.34) also indicated the addition of H_2O_2 improves the removal efficiency.

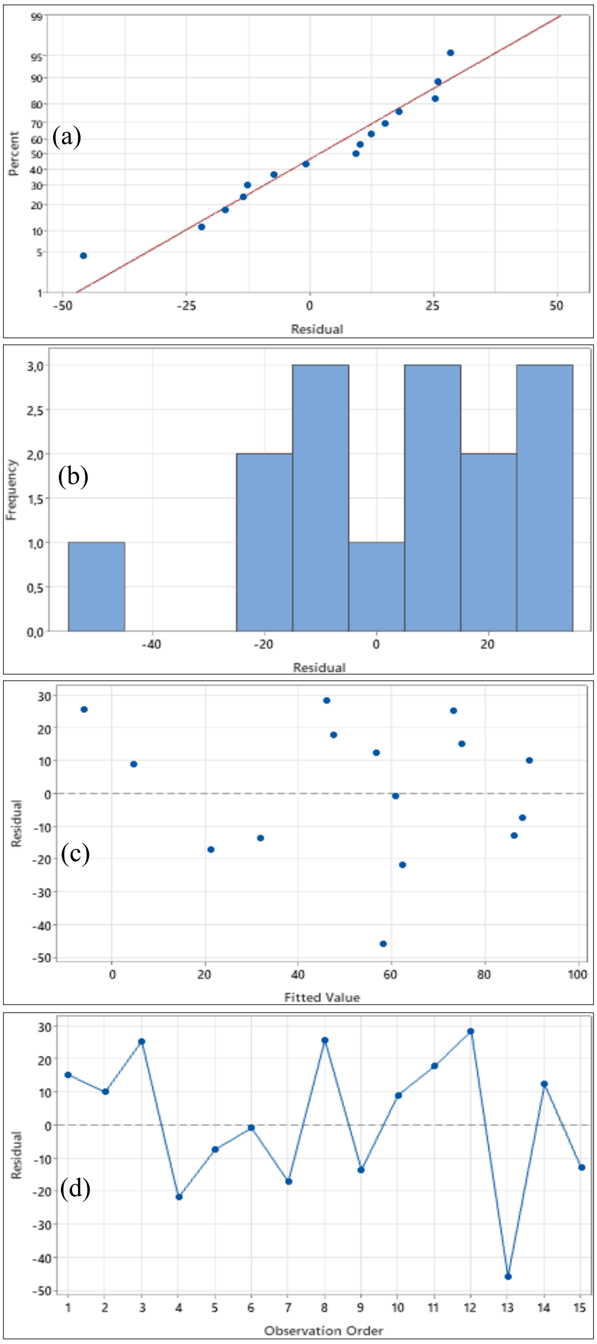

Figure 1. (a) Normal probability plot of residuals, (b) Histogram, (c) Residuals versus fits, (d) Residuals versus observation order

Pareto chart is the graphical representation on which factors have significant effects on the removal of PO_4-P. The related chart (Figure 2) also confirmed the outcomes derived from p values and coefficients of input parameters in regression analysis. H_2O_2 is the powerful component that positively effects the removal efficiency depending on the critical value above 2.228, which is indicated as the redline on Figure 2. The terms of NH_4, NH_4^2 and NH_4^3 can be interpreted as being close to the critical value of 2.228. Thus, NH_4 could be taken as a component that have effect on the removal efficiency. The effect of Na_2S can be regarded as limited due to the standardized effect below the critical value. However, Na_2S is a compulsory component in the synthesis of ZnO_xS_y particles. Thus, its effect is limited in the concentration range applied during synthesis.

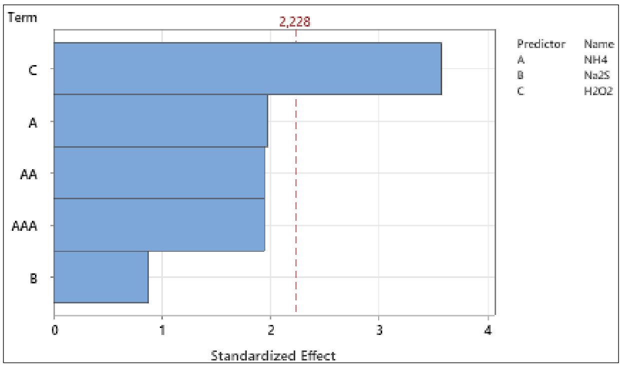

Figure 2. Pareto chart of the standardized effects

4. Conclusion

ZnO_xS_y nanoparticles were synthesized using different proportions of components and PO_4-P removal performance was tested. The PO_4-P removal efficiency was 99.5% when the initial PO_4-P concentration was 15 mg/L, adsorbent dose: 1 g/L, pH: 4.31, within a contact time of 2 hrs. The removal of PO_4-P was modelled by regression analysis with a high R^2 of 89.61% and p value 0.000. The main component that positively affect the removal performance was H_2O_2 by considering the statistical meaning. NH_4 component was close to the borderline of the statistical meaning, which indicated the importance of this component. Even though Na_2S component was found not to have statistically meaning, it should be noted that the model was composed and run in a pre-determined value.

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The Declaration of Conflict of Interest/ Common Interest

No conflict of interest or common interest has been declared by the author.

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