

Comparative Assessment of Phosphorus Removal from Nigeria's Agbaja Iron Ore Using Potassium Chlorate and Sodium Hydroxide

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Abstract

This paper presents a comparative assessment of phosphorus removal from Nigeria's Agbaja iron ore using powdered potassium chlorate (KClO₃) and sodium hydroxide solution (NaOH). Multifactorial influence of input process parameters such as treatment temperature, treatment time and concentration of leachants on phosphorus removal were studied within the ranges: 400-750⁰C, 600-4800 secs., and 9-16g respectively. Statistical analysis of results of phosphorus removal generated by using KClO₃ and NaOH were carried. The mechanism of the dephosphorization process was also investigated. Linear regression analysis was used to predict the trend of the results of phosphorus removal using KClO₃ and NaOH. These results were compared with the respective experimental results to level of precision. Results of the investigation on the effects of treatment temperature, treatment time and concentration of leachants on phosphorus removal indicated that in all the removed phosphorus concentration was higher on using NaOH compared to usage of KClO₃. Statistical analysis shows that the standard errors incurred during experiment in removing phosphorus relative to considered treatment temperatures, treatment times and concentrations of leachants for KClO₃ and NaOH are 0.3138, 1.0054 & 0.3025 and 0.8672, 3.5407 & 1.4442 respectively. The correlations between phosphorus removal and each of treatment temperature, treatment time and concentration of leachants used was higher for KClO₃ (0.9672, 0.8910 & 0.9746) compared to NaOH (0.8901, 0.7624 & 0.7624). This was attributed to the direct attack of oxygen (from KClO₃ decomposition) on the ore, unlike in the case of NaOH where oxygen emerges from a second reaction involving decomposition of hydrogen peroxide (H₂O₂) before attacking the ore. The mechanism of the dephosphorization process using KClO₃ and NaOH was strongly believed to be gaseous combination of oxygen gas (from decomposition of KClO₃ and H₂O₂ respectively) and phosphorus which transformed to vapour within the Gas Evolution Transition Range (GETR); 400-500⁰C. Results predicted using Linear Regression Analytical Method show significantly proximate and similar trend of data point's distribution relative to experimental results.

Keywords: Comparative Assessment, Phosphorus Removal, Agbaja-Nigeria Iron Ore, Potassium Chlorate and Sodium Hydroxide

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INTRODUCTION

The need for a drastic reduction in the concentrations of phosphorus present in iron ore as a way of upgrading the Fe content of the ore has necessitated a lot of researches. This is a step in a positive direction in actualizing the production of iron and steel materials that can withstand high stress and strain without possibly failing in service due to brittleness.

Successful attempt [1] has been made in the past to remove phosphorus from Changde iron ore. This process involves alkali-leaching and acid-leaching. This research was carried out using sodium hydroxide, sulfuric, hydrochloric and nitric acids for leach solution preparation. The results of the investigation reveal that phosphorus occurring as apatite phase could be removed by alkali-leaching, but those occurring in the iron phase could not. Sulfuric acid was found to be the most effective among the three kinds of acid.

The iron ore was found [1] to contain an average of 1.12% phosphorus and after the dephosphorization process, 91.61% phosphorus removal was attained with 1% sulfuric acid after leaching for 20 min at room temperature, during which iron loss was found to be negligible; less than 0.25%. The pH value of solution after leaching with 1% sulfuric acid was about 0.86, which implied that acid was not exhausted during the process and it could be recycled. The dephosphorization process was made more economical due to the recycling of sulfuric acid solution.

The prospect of iron enhancement has been projected [2] following a successful establishment of an integrated treatment process proposed for the dephosphorization of iron ore. The process involves a treatment for the removal of the phosphorus from the ore by leaching and further processing of the leach solution. Phosphoric acid which is concentrated by evaporation where most of the nitric acid is removed is extracted by isoamyl alcohol (iAmOH) and stripped by nitric acid solution during this process. The remaining nitric acid is extracted by methyl isobutyl ketone. The technical feasibility of the process was established and the economy analyzed and found viable in the sense that raffinate from the phosphoric acid extraction is treated by sulfuric acid for the regeneration of the spent nitric acid, which is extracted by iAmOH and concentrated by distillation before re-used in further leaching.

The aim of this work is to comparatively assess phosphorus removal from Nigeria's Agbaja iron ore using powdered potassium chlorate ($KClO_3$) and sodium hydroxide solution (NaOH) with the view to ascertaining the best amongst the chemicals.

MATERIALS AND METHODS

Agbaja (Nigeria) iron ore concentrate used for this work was obtained from Nigeria Metallurgical Development Centre (NMDC) Jos. This concentrate was dried in air (under atmospheric condition) and used in the as-received condition with particle size; $150\mu m$. A weighed quantity of the dried iron ore concentrate was mixed with different proportions of powdered $KClO_3$ (obtained from Fisher Scientific Company Fair Lawn, New Jersey, USA) as weighed with a triple beam balance at NMDC laboratory. Iron crucibles were filled with the sample mixtures of 9g of $KClO_3$ and 50g of ore concentrate. These samples in the crucibles were then heated to a temperature of $400^\circ C$ in a Gallenkamp Hot pot electric furnace at NMDC Laboratory for 600 secs. and thereafter were emptied on white steel pans for observation. The experiment was repeated using varied combination of mass-input of $KClO_3$ i.e 10, 11, 12, 13, 14, 15 & 16g and treatment temperature i.e 450, 500, 550, 600, 650, 700 and $750^\circ C$, while the mass-input of the ore is kept constant. Weighed quantities of the sample mixtures for each experiment set were taken (after being heated) for chemical analysis (to determine percentage phosphorus removal) using wet analysis method. The average of the phosphorus removed concentration determined in each experiment set was taken as the precise result. The whole experimental strategy was repeated for sodium hydroxide usage.

RESULTS AND DISCUSSIONS

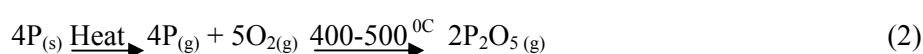
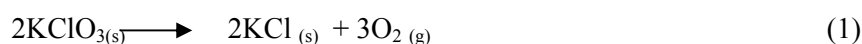
The result of the chemical analysis carried out on the beneficiated iron ore concentrate is presented in Table 1. The table shows that the percentage of phosphorus present in the as-beneficiated ore is 0.9%.

Table 1: Result of chemical analysis of iron ore used [3]

Element/Compound	Fe	P	SiO ₂	Al ₂ O ₃
Unit (%)	78.6	0.90	5.30	11.0

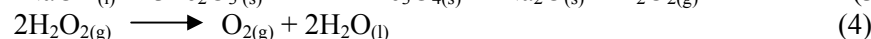
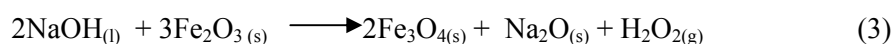
Mechanism of Dephosphorization

It is strongly believed that the mechanism of the dephosphorization process using KClO₃ and NaOH was gaseous combination of oxygen gas (from decomposition of KClO₃ and H₂O₂ respectively) and phosphorus which transformed to vapour within the Agbaja iron ore Gas Evolution Transition Range (GETR); 400-500^oC. Oxygen gas produced from equation (1) reacts with phosphorus to produce and give off P₄O₁₀ as shown in equation (2) in accordance with past findings [4] which posited that phosphorus sublimates between 400 – 500^oC to P₄O₁₀ in air.



At this temperature range, the reaction between phosphorus and oxygen moves with ease since both are in gaseous state.

Equation (3) shows a reaction between NaOH and the iron ore (Fe₂O₃). This reaction produced hydrogen peroxide gas which then decomposed to produce oxygen gas (in agreement with past findings [5]). The oxygen gas oxidized phosphorus which had already been transformed to vapour within the GETR and was removed in the form P₂O₅ as shown in equation (5). The possibility and viability of these reactions (equations (3)-(5)) stemmed from past work [6] where KOH was used as the leachant and sulphur was significantly removed in place of phosphorus.



Effects of treatment temperature, treatment time and mass-input of leachant on phosphorus removal

Results of the investigation on the effects of treatment temperature, treatment time and mass-input of leachants on phosphorus removal indicated (as in Figs.1 - 3) that in all, the removed phosphorus concentration was higher on using NaOH compared to usage of KClO₃.

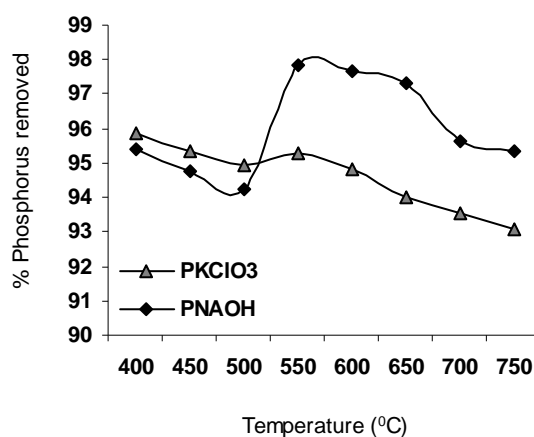


Fig. 1: Comparison of removed phosphorus concentration (relative to treatment temperature) as obtained from KClO₃ and NaOH [3].

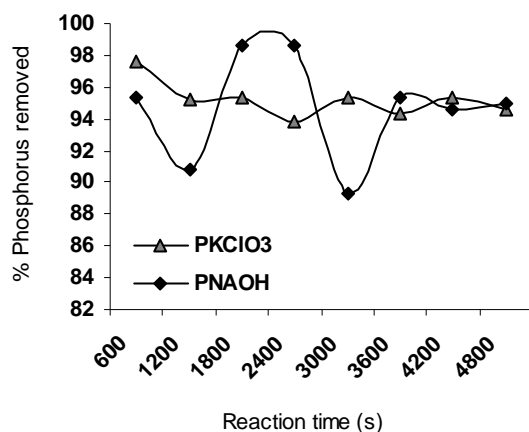


Fig. 2: Comparison of removed phosphorus concentration (relative to reaction time) as obtained from KClO₃ and NaOH [3].

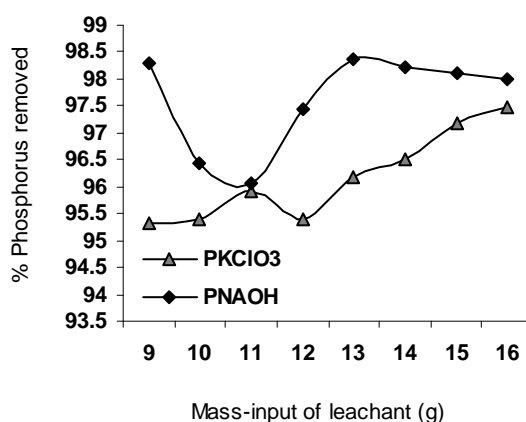


Fig. 3: Comparison of removed phosphorus concentration (relative to mass-input of leachant) as obtained from KClO₃ and NaOH [3].

Statistical analysis

Standard error

The standard errors (STEYX) in predicting the removed phosphorus concentration for each value of treatment temperature, treatment time and mass-input of leachant were evaluated as obtained from KClO₃ and NaOH. The evaluated error values are shown in Table 2. Table 3 shows evaluations from some analytical tools which indicate proximity between results generated from KClO₃ and NaOH. These evaluations were carried out using Microsoft Excel 2003 version.

Table 2: Comparison of standard errors associating experimental results from KClO₃ and NaOH [3]

STEYX	KClO ₃	NaOH
P & Leachant	0.3138	0.8672
P & Time	1.0054	3.5407
P & Temp	0.3025	1.4442

Table 3: Comparison of evaluations from selected analytical tools as obtained from experiments using powdered KClO₃ and NaOH solution [3]

Analysis	KClO ₃	NaOH
DEVSQ	1028096.56	1022705.225
AVEDEV	240.1939	239.4906
SKEW	0.3212	0.3228

Correlations

The correlations between removed phosphorus concentration and each of treatment temperature, treatment time and mass-input of leachant (as shown in Figs. 4-9) were evaluated from experimental results associating KClO_3 and NaOH considering the coefficient of determination R^2 also from Figs. 4-9 using the equation;

$$R = \sqrt{R^2} \quad (6)$$

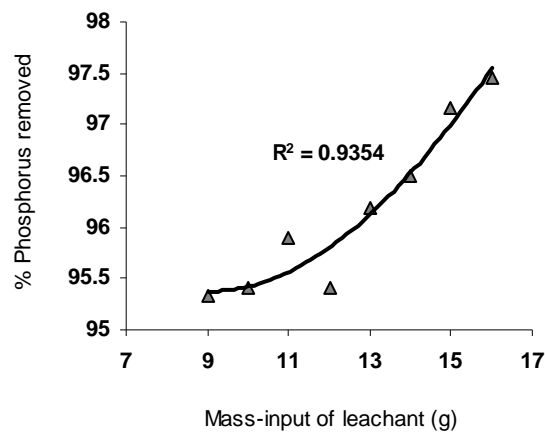


Fig. 4: Coefficient of determination between removed phosphorus concentration and mass-input of KClO_3 [3].

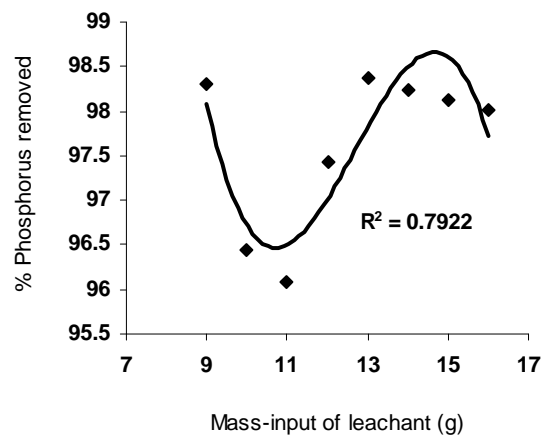


Fig.5: Coefficient of determination between removed phosphorus concentration and mass-input of NaOH [3].

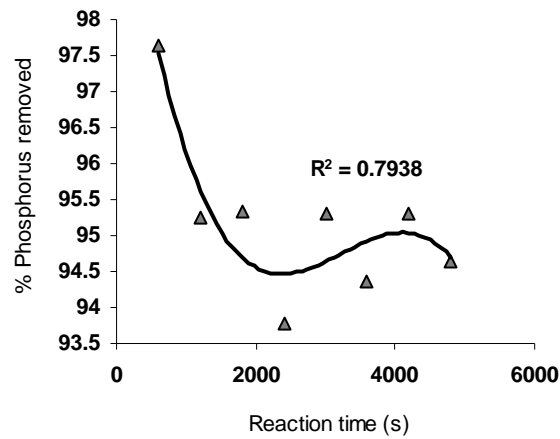


Fig. 6: Coefficient of determination between removed phosphorus concentration and reaction time as obtained from KClO₃ [3].

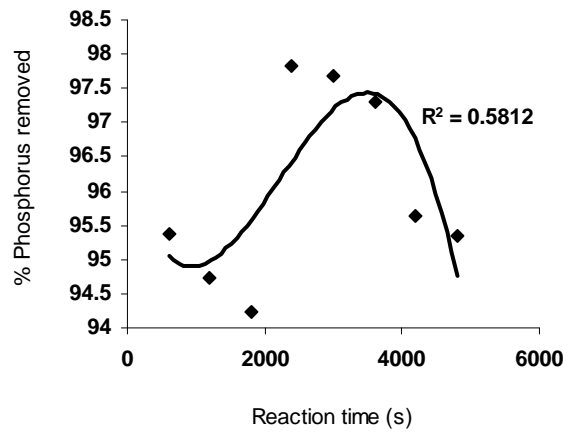


Fig. 7: Coefficient of determination between removed phosphorus concentration and reaction time as obtained from NaOH [3].

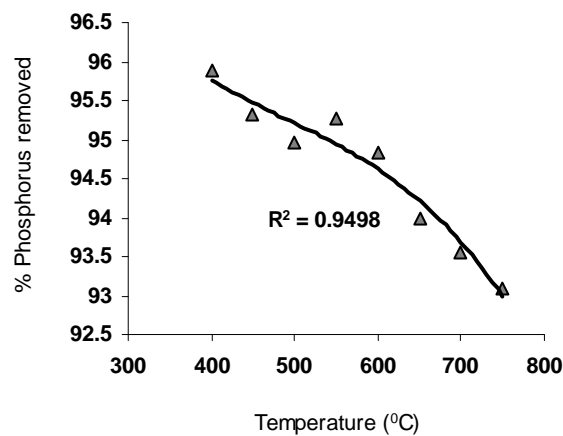


Fig. 8: Coefficient of determination between removed phosphorus concentration and treatment temperature as obtained from KClO₃ [3].

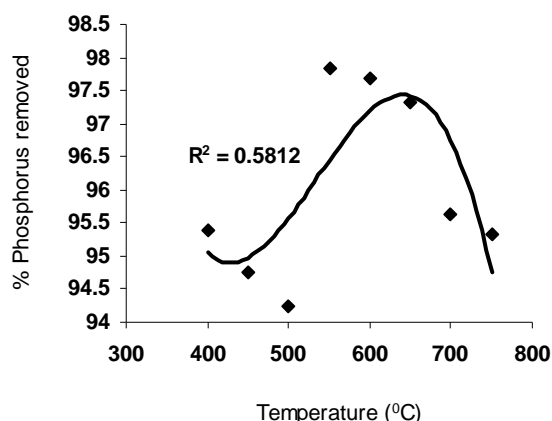


Fig. 9: Coefficient of determination between removed phosphorus concentration and treatment temperature as obtained from [3].

The evaluated correlations are shown in Table 4 for comparison. The correlations between phosphorus removal and each of treatment temperature, treatment time and concentration of leachants used was higher for $KClO_3$ compared to $NaOH$. This was attributed to the direct attack of oxygen (from $KClO_3$ decomposition) on the ore, unlike in the case of $NaOH$ where oxygen emerges from a second reaction involving decomposition of hydrogen peroxide (H_2O_2) before attacking the ore.

Table 4: Comparison of correlations associating experimental results from $KClO_3$ and $NaOH$ [3]

Correlation	$KClO_3$	$NaOH$
P & Leachant		
P & Time	0.9672	0.8901
P & Temp.		

Comparison of experimental results of $KClO_3$ and $NaOH$ with standard model prediction

The precision of the experimental work was further verified through application of the Linear Regression Analysis (LSM) which predicted the trend of the experimental results. Comparative analysis of Figs. 10-15 shows very close alignment of curves and significantly similar trend of data point's distribution for experimental results obtained using $KClO_3$ and $NaOH$.

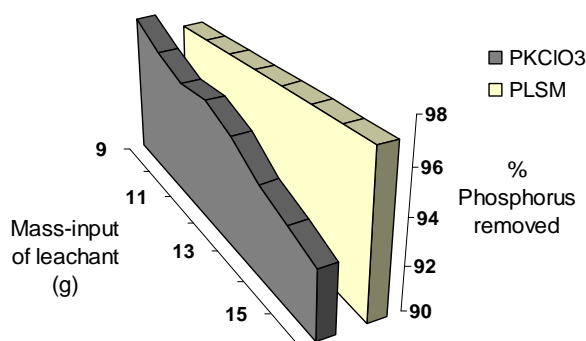


Fig. 10: Comparison of removed phosphorus concentration (relative to mass-input of leachant) as obtained from $KClO_3$ [3] and LSM.

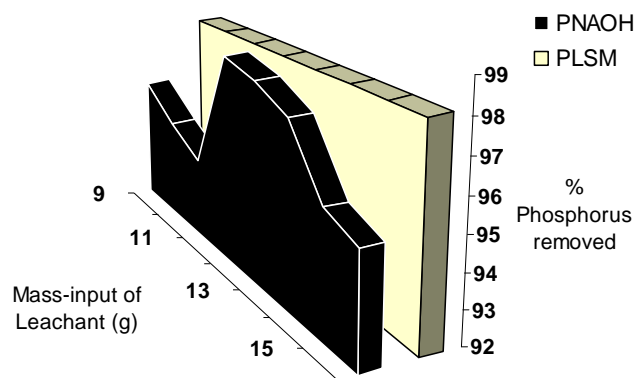


Fig. 11: Comparison of removed phosphorus concentration (relative to mass-input of leachant) as obtained from NaOH [3] and LSM.

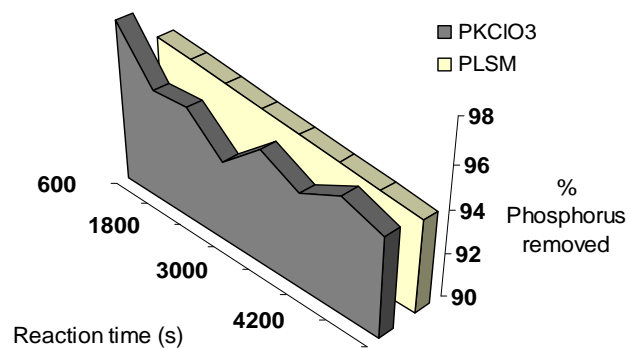


Fig. 12: Comparison of removed phosphorus concentration (relative to treatment time) as obtained from KClO₃ [3] and LSM.

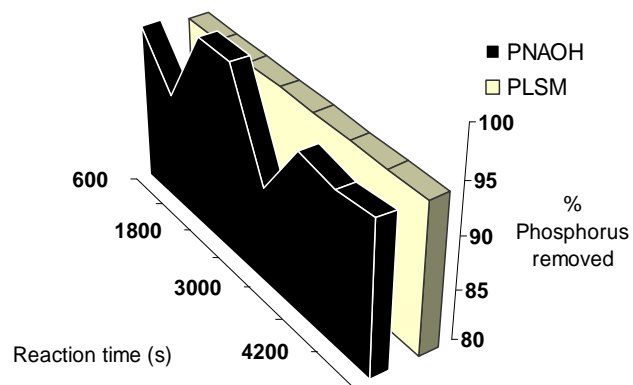


Fig. 13: Comparison of removed phosphorus concentration (relative to treatment time) as obtained from NaOH [3] and LSM.

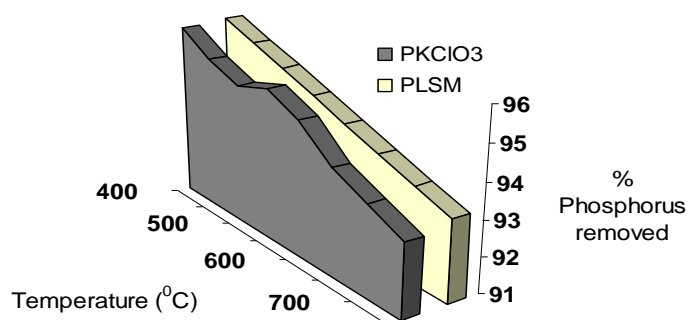


Fig. 14: Comparison of removed phosphorus concentration (relative to treatment temperature) as obtained from KClO₃ [3] and LSM.

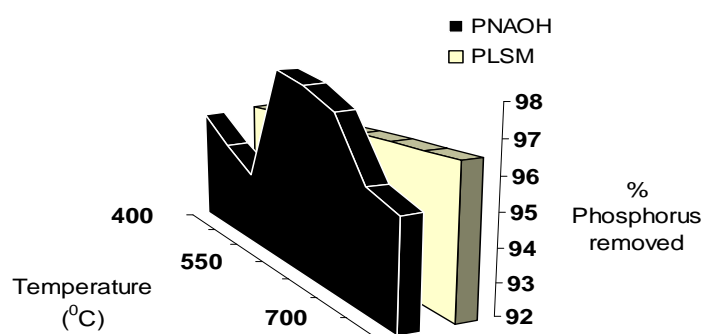


Fig. 15: Comparison of removed phosphorus concentration (relative to treatment temperature) as obtained from NaOH [3] and LSM.

CONCLUSION

Results of the investigation on the effects of treatment temperature, treatment time and concentration of leachants on phosphorus removal indicated that in all the removed phosphorus concentration was higher on using NaOH compared to usage of KClO₃. From statistical analysis, the standard errors incurred during experiment in removing phosphorus relative to considered treatment temperatures, treatment times and concentrations of leachants for KClO₃ and NaOH were 0.3138, 1.0054 & 0.3025 and 0.8672, 3.5407 & 1.4442 respectively. The correlations between phosphorus removal and each of treatment temperature, treatment time and concentration of leachants used was higher for KClO₃ (0.9672, 0.8910 & 0.9746) compared to NaOH (0.8901, 0.7624 & 0.7624). This was due to the direct attack of oxygen (from KClO₃ decomposition) on the ore, unlike in the case of NaOH where oxygen emerges from a second reaction involving decomposition of hydrogen peroxide (H₂O₂) before attacking the ore. The mechanism of the dephosphorization process using KClO₃ and NaOH involved gaseous combination of oxygen gas (from decomposition of KClO₃ and H₂O₂ respectively) and phosphorus which transformed to vapour within the Gas Evolution Transition Range (GETR); 400-500°C. Results predicted using Linear Regression Analytical Method show significantly proximate and similar trend of data point's distribution relative to experimental results.

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