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Optimization of flotation efficiency of phosphate minerals in mine tailings using polymeric depressants: Experiments and machine learning

Ashraf Alsafasfeh [1,2], Lana Alagha [1,3], Ala Alzidaneen [4], Venkata Sriram Siddhardh Nadendla [5]

[1] Department of Mining and Explosives Engineering, Missouri University of Science and Technology, MO65409, USA
[2] Department of Natural Resources and Chemical Engineering, Tafila Technical University, Tafila, Jordan 66110
[3] Thomas J. O'Keefe Institute for Sustainable Supply of Strategic Minerals, Rolla, MO, USA, 65409, USA
[4] Department of Electrical and Computer Engineering, Missouri University of Science and Technology, Rolla, MO, USA
[5] Department of Computer Science, Missouri University of Science and Technology, Rolla, MO, USA

Corresponding author: aalagha@umsystem.edu (Lana Alagha)

Abstract: In this study, direct froth flotation experiments were conducted on silicate-rich phosphate tailing samples. The average grade of P_2O_5 in the flotation feed was 21.6% as determined using a combination of spectroscopic techniques including X-ray powder diffraction (XRD), mineral liberation analysis (MLA), and scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDS). Two polymers were selected to promote the depression of silicates and enhance the flotation of phosphates: in-house synthesized hybrid polyacrylamide (Hy-PAM) and chitosan. Flotation efficiency of phosphates was evaluated at different flotation conditions including depressant type, depressant dosage, pH, and the flotation time. Results indicated that the optimum flotation efficiency of phosphate minerals (84.6% recovery at 28.6% grade of P_2O_5) was obtained when Hy-PAM was utilized at the studied range of pH and flotation time. All datasets produced from the flotation experiments were integrated within the framework of machine learning (ML) using artificial neural networks (ANNs). The ANN platform was trained, validated, and successfully employed to predict the process outcomes in relation to the pulp and reagents characteristics, which in turn were used to determine the optimum values of process variables. Coefficient of determination (R^2), mean absolute error (MAE), and root-mean-square error (RMSE) were used as model indicators. Optimization results showed that the peak flotation performance could be achieved at higher dosages of both polymers. However, lower pH and shorter flotation time for Hy-PAM, and higher pH and longer flotation time for chitosan, were predicted to give the optimum process efficiency.

Keywords: phosphate tailing, froth flotation, polymers, chitosan, artificial neural networks

1. Introduction

Mineral tailings produced from phosphate processing plants are considered the main secondary sources of phosphate minerals and can be enriched to produce concentrates that meet the requirements to produce phosphate fertilizers. Phosphate minerals in tailings are usually found associated with fine and ultrafine gangue minerals such as silicates and carbonates, which makes their separation and enrichment very challenging (Alsafasfeh, 2020; Oliveira et al., 2011). Froth flotation process has proven to be one of the best methods that allows efficient separation of minerals, especially when dealing with lower grade ores and thus can be applied to upgrade phosphate tailings. Froth flotation process utilizes the differences in surface wettability of different minerals at water-solid-gas interfaces so that hydrophobic minerals attach to air bubbles and float while hydrophilic minerals report to tailings (Chen et al., 2003; Kyzas & Matis, 2019; Zhang, 2013). Froth flotation technique is usually used to upgrade phosphate ores in most phosphate production plants due to its high selectivity and versatility. In this

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practice, phosphate minerals can be separated from the associated gangue minerals (silicates, carbonates, etc.) using either direct flotation process (phosphates are floated) or reverse flotation process (gangues are floated) (Al-Thyabat et al., 2011; El-Shall et al., 2004; Peleka et al., 2006). The best method is selected based on the mineral composition of the ore (El-Shall et al., 2004; Yehia et al., 1990). In general, the grade of phosphates in the ore and flotation concentrates is expressed in terms of phosphorus pentoxide (P_2O_5), the major precursor to produce phosphoric acid-based fertilizers. The grade of P_2O_5 in flotation concentrate products should be at least 28-30% for economic production of phosphate fertilizers, (Alsafasfeh & Alagha, 2017; Holmes et al., 1982).

Despite the extensive and successful application of flotation process in phosphate separation and enrichment, selective removal of silicate minerals remains a serious challenge. This is primarily because these minerals are usually liberated at fine and ultrafine sizes, especially in lower grade ores and tailings produced from phosphate processing (important secondary sources of phosphates) (Alsafasfeh & Alagha, 2017; J. C. Liu et al., 2009). Therefore, they tend to entrain into the froth layer during flotation and deteriorate the grade of phosphate concentrates (Boulos et al., 2014; X. Liu et al., 2016). Development of selective, flexible, and green chemical reagent systems is considered as one of the most effective approaches to address this challenge. Although common inorganic depressants that are commonly used in phosphate flotation practices (such as sodium silicates) have shown good performance in depressing the flotation of silicate minerals, recent studies indicated that sodium silicate is toxic to aquatic and terrestrial organisms (Of & Products, 2005; Van Dokkum et al., 2004). Moreover, it may cause irritation to the skin and eyes (*National Library of Medicine HSDB Database*, 2019). Therefore, its replacement with green reagents from sustainable resources is appealing.

Among the different types of chemical reagents, biocompatible polymeric materials (natural, synthetic, and hybrid) have received significant attention. Polymers have been successfully applied as multifunctional reagents in phosphate flotation processes due to their biocompatibility, tunability, selectivity, and relatively lower cost. For example, starch and guar gum have been used as phosphates depressants in the reverse flotation process of phosphate and iron ores (Nagaraj et al., 1987; Nanthakumar et al., 2009). Polyepoxysuccinic acid, was successfully applied as a selective depressant of calcite in calcite/apatite flotation in the presence of sodium oleate collector (Dong et al., 2021). Hybrid polyacrylamide polymers were used as silicate depressants in direct flotation of phosphate ores, and preliminary results indicated that this polymer enhanced the recovery and grade of phosphate concentrates at specific conditions (Alsafasfeh et al., 2018; Khodakarami & Alagha, 2017). Motivated by the ongoing research efforts to use flexible and yet biocompatible reagents in phosphate flotation practices, this study proposed two different types of polymeric depressants to allow selective separation of silicates from phosphates: chitosan as an example of natural biodegradable polymers and hybrid polyacrylamide (Hy-PAM) as an example of functionalized biocompatible polymer. The structures of both polymers are illustrated in Fig. 1. Hy-PAM is an organic-inorganic polymer that consists of polyacrylamide organic chains grafted on nano-size inorganic Al $(OH)_3$ particles. A previous study reported the capability of this polymer to adsorb on ultrafine silica particles and promote their flocculation (Alagha et al., 2011). In addition, Hy-PAM was successfully applied in fine coal flotation to enhance the combustible recovery and reduce the ash contents (mainly fine clays and silicates) of coal concentrates (Molatlhegi and Alagha, 2016). Preliminary investigations also indicated that this polymer had the potential to depress silicates in phosphate flotation (Alsafasfeh, 2020). Chitosan, on the other hand, has shown a strong affinity to silicate minerals and was proven effective in flocculation of clays, silicates, and quartz suspensions (Bina et al., 2013; Zemmouri et al., 2012). Thus, both polymers have the potential to serve as selective depressants of silicates in phosphate flotation.

In addition to the type of chemical reagents, the separation efficiency of phosphates from silicates by the froth flotation process is impacted by several other variables. Among these are the physicochemical factors such as pulp pH and salinity; and the operational factors such as agitation rate in the mechanical flotation cells, air flowrate, solid concentration, mineralogical composition, the particle size distribution of feed, and flotation time (Kawatra, S. Komar, 2013; Klimpel, 1995; Sis & Chander, 2003). The mutual interaction among these variables is complex and poses a challenge for process control and optimization (Ali et al., 2018). Thus, the development of adaptive and intelligent control systems to predict and optimize the separation of phosphates from silicates by froth flotation, especially when new reagents are utilized, is of central importance. This study applied machine learning to process the experimental data produced from flotation tests to predict and optimize the flotation performance of phosphate minerals when chitosan and Hy-PAM were used as silicate depressants.

Machine learning (ML) and artificial intelligent (AI) models have been recently used to predict and optimize the efficiency of the froth flotation process in many applications. Multi-layered artificial neural networks (ANN) and random forests (RF) models were used based on froth image analysis to estimate concentrate grade in platinum flotation (R. Peter King, 2012). Jorjani used ANN to predict sulfur's separation efficiency in coal flotation (Jorjani et al., 2008). Multi-layered ANN was successfully employed to predict clayey coal's flotation behavior in the presence of novel ash depressant ($Al(OH)_3$-PAM polymer) at five process variables (Khodakarami et al., 2019). A comparison study of five different models (Mamdani fuzzy logic, hybrid neural fuzzy inference system, adaptive neuro-fuzzy inference system, RF, and ANN) was performed by Ali and others to predict the flotation performance of high-ash coal (Ali et al., 2018). Yafeng et al. (2020) predicted the flotation performance of magnesium-bearing carbonate ore at different size fractions using random forest (RF), extra tress (ET), and artificial neural network (ANN) (Fu et al., 2020). Cook et al. (2020) employed an original hybrid ML model - RF-FFA, developed by integrating RF model and the firefly algorithm (FFA) – to predict froth flotation efficiency of galena and chalcopyrite, from a complex sulfide ore sample, in relation to various experimental process parameters. In phosphate flotation process, Gouws and Aldrich used probabilistic induction and genetic algorithms to predict the flotation efficiency of phosphates by analyzing the structures of the froth layers (Gouws & Aldrich, 1996). Al-Thyabat used ANN to predict the effect of flotation variables including collector dosage, feed size, and impeller speed on the flotation efficiency of phosphates from siliceous phosphate ore (Al-Thyabat, 2008). Due to its high versatility and successful application in prediction of flotation behavior of minerals in many complex flotation systems, ANN model was chosen in this study to predict and optimize the flotation efficacy of phosphate minerals.

The major goals of this study were to: (i) improve flotation efficiency of phosphate minerals from phosphate mine tailings by utilizing novel and selective silicate's depressants (Hy-PAM and chitosan) and (ii) predict and optimize the flotation performance of phosphate minerals in the presence of Hy-PAM and chitosan using artificial neural networks (ANN). For this purpose, direct froth flotation experiments were conducted, at bench scale, on silicate-rich phosphate tailing samples. Flotation efficiency of phosphate minerals was evaluated at different flotation conditions. All datasets produced from the flotation experiments were assimilated within the progressive-and-adaptive framework of machine learning (ML) using artificial neural networks (ANN). The training and validation process of the ANN platform was employed to predict the flotation outcomes (phosphate recovery and concentrate grade expressed as $\%P_2O_5$) in relation to variables related to pulp and reagents characteristics (flotation time, pulp's pH, depressant dosage, and depressant type). Results obtained from the developed ANN model were used, thereafter, to optimize the flotation performance of phosphate minerals.

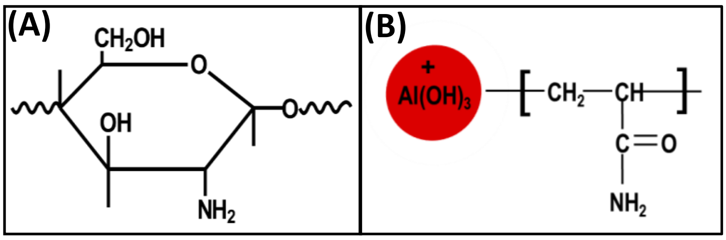

Fig. 1. The chemical formula of (A) Chitosan polymer, and (B) hybrid polyacrylamide polymer (Hy-PAM)

2. Materials and methods

2.1. Materials

Phosphate tailing samples used in this study were obtained from a phosphate plant located in the North America and characterized as described in the following section. All chemical reagents used in the flotation tests, except for hybrid polyacrylamide (Hy-PAM), were purchased from Fisher Scientific, USA. This included sodium silicate (a commercial depressant of silicate minerals), sodium oleate (phosphate collector), methyl isobutyl carbinol (MIBC, frother), and chitosan polymer (proposed silicate's depressant). Hydrochloric acid (HCl) and sodium carbonate (Na_2CO_3) that were used as pH modifiers were also purchased from Fisher Scientific. The reported molecular weight and deacetylation degree of chitosan polymer were 1526.46 g/mol and 85%, respectively. On the other hand, Hy-PAM (proposed silicate's depressant) was synthesized in-house according to a procedure described in a previous research work (Alagha et al., 2011; Alsafasfeh et al., 2018; O. Molatlhegi et al., 2015). In summary, Hy-PAM was prepared by free radical polymerization of acrylamide monomers in $Al(OH)_3$ nano suspensions that have a particle size of ~ 30-50 nm and zeta potential value of ~ +27-30 mV. The aluminum content of the synthesized Hy-PAM was 0.14 wt.% as determined using PerkinElmer inductively coupled plasma system equipped with optical emission spectrophotometer (ICP-OES). The molecular weight of Hy-PAM polymer was $6*10^6$ g/mol as determined using the Zetasizer Nano ZS instrument.

2.2. Characterization of the phosphate tailing samples

Extensive characterization studies were conducted on phosphate tailing samples using X-ray powder diffraction (XRD), mineral liberation analysis (MLA), and scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDS). As per industrial procedures, the size range of +35 -125µm, obtained by sieving, was used as flotation feed (Santana et al., 2008). MLA results indicated that apatite and fluorapatite were the primary phosphorus-containing phases in the feed samples while the main gangue mineral was quartz which constituted 17.2% of the sample. As determined by MLA, the grade of P_2O_5 in the feed sample was ~ 24.8%. Moreover, liberation analysis of the flotation feed indicated that apatite minerals were better liberated than quartz, as shown in Supplementary Information (SI), Section SI.1, Fig. S1. SEM/EDS analysis (SI.1, Table S1) revealed the presence of O, F, Ca, P, and Si atoms in large amounts, which demonstrated that apatite, fluorapatite, and quartz are the dominant minerals in the flotation feed. The grade of P_2O_5 in the feed as determined by SEM-EDS was 17.5%. XRD results showed that the feed sample contained 22.6% of P_2O_5. Therefore, the average grade of P_2O_5 in the feed was determined to be ~21.6% which was calculated based on the results obtained from MLA, SEM/EDS, and XRD.

2.3. Zeta potential measurements

To get fundamental insights into the adsorption selectivity of the proposed polymeric depressants on mineral surfaces, the electrical properties of model apatite and quartz suspensions, before and after treatment with Hy-PAM and chitosan, were examined using zeta potential measurements. Zeta potential measurements were performed using a Zetasizer Nano ZS by mixing 0.1 wt.% pure mineral in 0.1M potassium chloride (KCl) (Alsafasfeh et al., 2018; Alsafasfeh & Alagha, 2017). The pH of the prepared stock solutions was adjusted as needed using sodium carbonate (Na_2CO_3) and hydrochloric acid (HCl). More details on sample preparation for zeta potential measurements have been added to **SI, Section SI.2**.

2.4. Froth flotation experiments

A laboratory-scale Denver flotation machine was used in all flotation experiments. Baseline (control) experiments were first conducted to define the base flotation performance of phosphates (recovery and grade) using sodium oleate and MIBC without the addition of any depressant. All other depressants were tested, thereafter, at different flotation conditions (depressant type, depressant dosage, pH of the flotation pulp, and the flotation time). Table 1 shows the experimental conditions and parameters used in this study. A flowchart of the flotation experimental procedures used in this study is shown in SI, Section SI.3, Fig. S2. The pulp containing the flotation feed (+35-125 micron) in tap water was first agitated for 4 minutes, followed by the addition of sodium carbonate to adjust the pH as needed. Sodium oleate (phosphate's collector) was then added, followed by silicate's depressant (sodium silicate, Hy-PAM, or chitosan) at a predetermined dosage as presented in Table 1. The pulp was agitated for 4 min then MIBC was added. The concentrate products were collected at 4 min and 10 min flotation time, dried, and assayed for P_2O_5. The dry weights of both concentrates and tailing products were used to examine the flotation performance by measuring the recovery and grade of phosphate minerals using Eq. 1 where "C" is dry weight of the concentrate product (froth), "T" is dry weight of the tailing product, "c" is percentage of P_2O_5 in concentrate, and "t" is the percentage of P_2O_5 in tailing.

$$\text{Recovery} = Cc/\ (Cc+Tt) *100\% \tag{1}$$

Table 1. Experimental conditions and parameters tested in the flotation process of phosphate tailing

Variable conditions		Fixed conditions
Depressant type	Sodium silicates, Hy-PAM, and Chitosan	Solid percentage: 60 wt.% Collector dosage: 200 g/ton Frother dosage: 63 g/ton
Depressant dosage	0,150, 200, 250, and 300 g/ton	
Pulp pH	pH 7 and pH 9	
Flotation time	4 min and 10 min	

2.5. Datasets development and neural network design

The artificial neural networks (ANN) model was applied to predict and optimize the performance of phosphate flotation. ANN model is a very simplistic representation of how the human brain works. ANN is known for its high reputation in developing the algorithms that can be used to build a complex pattern for prediction problems as shown in Fig. 2 The ANN architecture has an input layer (one), hidden layer (one or more), and the output layer (one). Each layer consists of multiple processing units known as neurons or nodes. The nodes of one layer are connected with the nodes of subsequent layers and each connection has a factor known as weight. The impact of one node on another is determined through these connection weights (Ali et al., 2018). Input data are provided to the input layer which forwards the data to the the hidden layer and to the output layer wherein nodes process the data and apply weights to them. The weighted inputs are processed by each node by passing them through an activation function and the outputs arereleased (Cook et al., 2020; Hayat, 2018).

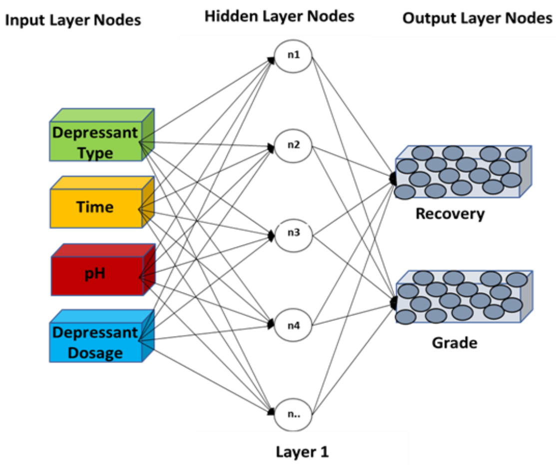

Fig. 2. A schematics of artificial neural network (ANN) architecture showing the input layer, the hidden layer, and the output layer

Experimental dataset produced from laboratory flotation tests of phosphate tailings (SI, Table S2) were used to train and test the performance of the ANN model. Flotation process parameters that were used as data inputs for the ANN model included the type and the dosage of silicate's depressant, the flotation time, and pulp's pH. Both recovery and grade of P_2O_5 were used as model's outputs. Fig. 3 shows the strategy applied to develop the ANN model using Python as it is considered an accessible programming language to develop ANN models (Hao & Ho, 2019; Hart et al., 2011).

Data cleaning function ***"isna()"*** was used to detect and remove the errors from the datasets in order to have reliable datasets and improve the model's performance. This function takes a scalar or array-like object and indicates whether values are missing. Then, based on the trial error method, the the dataset was split into 75% and 25% for training and testing, respectively, to evaluate the model

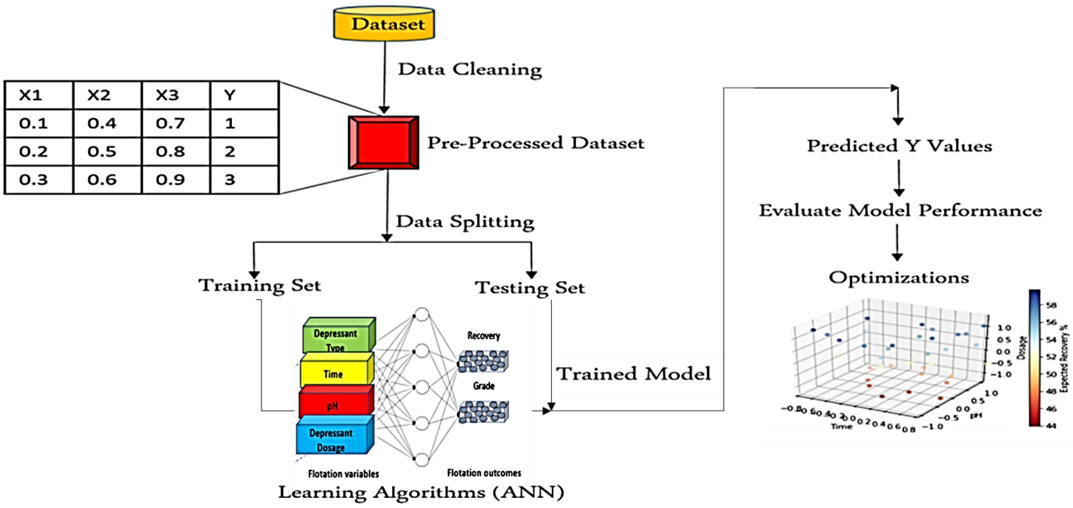

Fig. 3. Flowchart of the ANN strategy applied in this study: first datasets were developed wherein four process variables were used as input parameters. Datasets were then split for training and testing followed by model validation

(75%:25% gave better performance than 80%:20%, which also had more testing data for the validation process). The model was trained and validated using one-hidden layer, two hidden layers, and three-hidden layers at different number of nodes. The number of nodes were chosen based on trial-and-error method by comparing the values of R^2, MAE, and RMSE for both recovery and grade of phosphate minerals. For a single hidden layer, 20 nodes were used. For two hidden layers, 20 and 15 nodes were used. For three hidden layers 25,20,15 nodes were used. Mean absolute error "MAE", coefficient of determination "R^2", and root mean square error "RSME" were used as performance indicators of the ANN model (Eq. 2, 3 & 4). (Ali et al., 2018; Dou & Yang, 2018).

$$\text{MAE} = \frac{[\sum_{i=1}^{n}(yi-Xi)]}{n} \tag{2}$$

$$R^2 = \frac{[\sum_{i=1}^{n}(Yi-\bar{Y})(Xi-\bar{X})]^2}{[\sum_{i=1}^{n}(Yi-\bar{Y})^2(Xi-\bar{X})^2]} \tag{3}$$

$$RMSE = \sqrt{\frac{\sum_{i=1}^{n}(Yi-Xi)^2}{n}} \tag{4}$$

where, n = Number of datasets, Yi = Predicted value, Xi = Actual value, $\bar{Y}$ = Mean predicted values, $\bar{X}$ = Mean actual value.

Moreover, a 100 random data input for depressant dosage (in the range of 150g/ton to 300g/ton), pH (in the range of 7 to 11), and flotation time (in the range of 4 min to 10 min) were generated for each type of depressant used (i.e., sodium silicate, Hy-PAM, and chitosan). These data inputs were used thereafter to predict and optimize the flotation outputs (i.e., phosphate recovery and concentrate grade).

3. Results and discussion

3.1. Zeta potential measurement

Zeta potential of apatite and quartz suspensions (the two major minerals in the flotation feed) were measured before and after mixing with the proposed polymeric depressants (Hy-PAM and chitosan) at different pH to fundamentally explore the change of electrical properties of mineral suspensions and investigate the selectivity of reagents used in the flotation process. Fig. 4 (A and B) shows the changes in the magnitude of zeta potential values (Δζ) of quartz and apatite suspensions after the addition of different depressants at pH 7 and pH 9, respectively. As exhibited by the magnitude of Δζ, there was a stronger interaction (possibly electrostatic) between Hy-PAM and quartz surfaces compared to apatite surfaces at both pH values. For example, the changes in the magnitude of zeta potential values of quartz after mixing with Hy-PAM at pH 9 was around +12mV (from −34mV to −22mV) while it only increased by +6mV when Hy-PAM was mixed with apatite. Similarly, the change in the magnitude of zeta potential after mixing with chitosan was more significant for quartz compared to apatite at both pH values ($\Delta\zeta$ = ~+30 mV for chitosan-quartz system at both pH values). Results from zeta potential measurements suggested that Hy-PAM and chitosan polymers had a preferential adsorption on quartz surface compared to apatite. The stronger adsorption on quartz surfaces would make the surface more hydrophilic and thus reduce the probability of particle-bubble attachment which would result in flotation depression. In the light of these results, further investigation on the impact of chitosan and Hy-PAM on the flotation efficiency of silicate-rich phosphate tailing samples were conducted using batch flotation tests as described in the following sections.

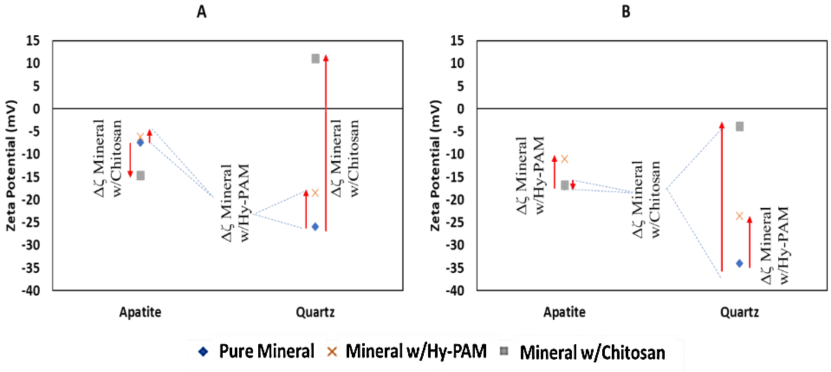

Fig. 4. Zeta potential of apatite and quartz suspensions before and after mixing with different chemical reagents at pH 7 (A) and pH 9 (B)

3.2. Froth flotation experiments

Flotation experiments on phosphate tailing samples were conducted under conditions presented in Table 1. As discussed, three different reagents were tested to suppress silicates/quartz flotation: sodium silicate as an example of inorganic commercial silicate's depressant, in-house synthesized hybrid polyacrylamide (Hy-PAM) as an example of functional synthetic polymers, and chitosan as an example of natural biodegradable polymers. Flotation process performance was expressed in terms of %recovery and % grade of P_2O_5.

3.2.1. Flotation experiments in the presence of sodium silicate depressant

Sodium silicate is considered one of the most effective inorganic depressants/dispersants for silicate minerals. Flotation experiments using sodium silicate were performed to allow comparison with polymeric depressants proposed in this study. In this set of experiments, different influencing parameters were examined to evaluate the flotation performance of phosphate minerals as listed in Table 1. As shown in Fig. 5, the higlest recoveries of phosphate minerals were observed at 250 g/ton sodium silicates and the pH doesn't have significant impact on the recoveries unlike the flotation time. For example, at 250 g/ton of sodluim silicates and pH 7 and pH 9, the recoveries of P_2O_5 were ~ 72%and 74%, respectively at 4 min flotation time. However, P_2O_5 recovery was 55.5% at pH 9 and 10 min of flotation time. This could be explained by the mechanism via which sodium silicates depresses minerals. It is anticipated that sodium silicate molecules hydrolyze in solution to produces monomeric, polymeric and colloidal species, depends on the pH, that adsorb on mineral surfaces and aid their flotation depression. At pH up to 9, the main soluble species is $Si(OH)_4$ and the concentration of this species is almost constant up to pH 9. The polymeric form of silicates has more depression effect, but it predominates at higher pH values. That is why the recoveries were almost similar at pH 7 and 9 at shorter time. At longer flotation time, the observed drop in recovery could be due to possible formation of negatively charged monomeric species in addition to $Si(OH)_4$ which will aid more depression of minerals (Qi et al., 1993; Althyabat, 2009; Kupka et al., 2020). Overall, the highest observed recovery and grade of P_2O_5 were 74% and 28.4%, respectively, at 4 min flotation time, 250 g/ton sodium silicate, and pH 9.

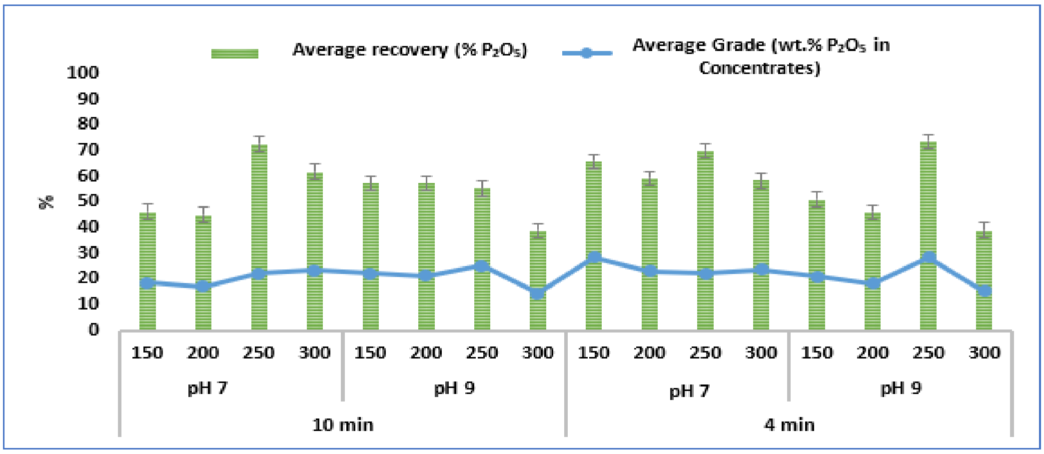

Fig. 5. Flotation efficiency of phosphate minerals in the presence of sodium silicate at different conditions

3.2.2. Flotation experiments in the presence of hybrid polyacrylamide (Hy-PAM)

The influence of Hy-PAM's dosage and the pulp's pH were examined at two different flotation times (4 min and 10 min). As shown in Fig. 6, at pH 7, the highest flotation recovery and grade of P_2O_5 were obtained when 250 g/ton of Hy-PAM was used at both shorter (4 min) and longer (10 min) flotation time. On the other hand, at pH 9, 300 g/ton of Hy-PAM was needed to achieve optimum recovery and grade. When comparing the flotation efficacy at a shorter flotation time, it was observed that the flotation recovery and grade showed the highest values at pH 7 and 250 g/ton of the polymer (84.6% recovery at 28.6% grade).

3.2.3. Flotation experiments in the presence of chitosan depressant

Fig. 7 shows the flotation outcomes (recovery and grade of P_2O_5) with chitosan at different flotation parameters. Good flotation performance of phosphates (67% recovery and 25% grade of P_2O_5) was obtained at longer time (10 min), pH 9, and 300 g/ton of chitosan, which indicated that increasing the flotation time, the pH, and chitosan dosage had a positive impact on the flotation efficiency of phosphate minerals.

Overall, the optimum flotation efficiency of phosphate minerals (peak recovery and grade) was obtained when Hy-PAM was utilized at a dosage of 250 and 300 g/ton at pH 7 and 9, respectively, and flotation time of either 4 or 10 min. At shorter flotation time of 4 min, 250 g/ton of depressant (typical dosage at industrial practices) and pH 7 (natural pH of tailing sample), the %recoveries/grades of P_2O_5 were ~ 81/28.4%, 70/22%, and 41/20% with Hy-PAM, sodium silicate, and chitosan, respectively. However, at pH 9 and similar flotation time and depressant's dosage, the recoveries/grades of P_2O_5 were ~ 65/27%, 74/28.4%, and 43/20% with Hy-PAM, sodium silicates, and chitosan, respectively. At longer flotation time of 10 min, pH 7, and 250 g/ton of depressant's dosage, the %recoveries/grades

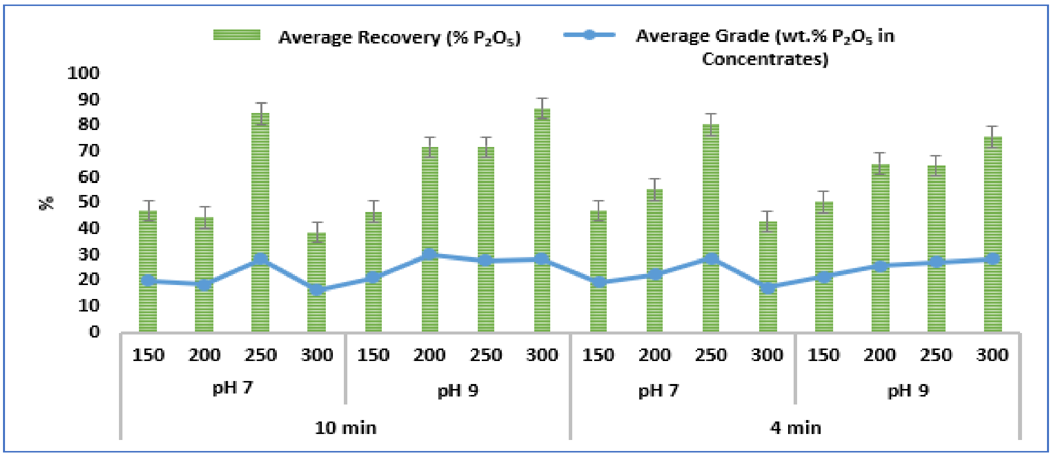

Fig. 6. Flotation efficiency of phosphate minerals in the presence of Hy-PAM at different conditions

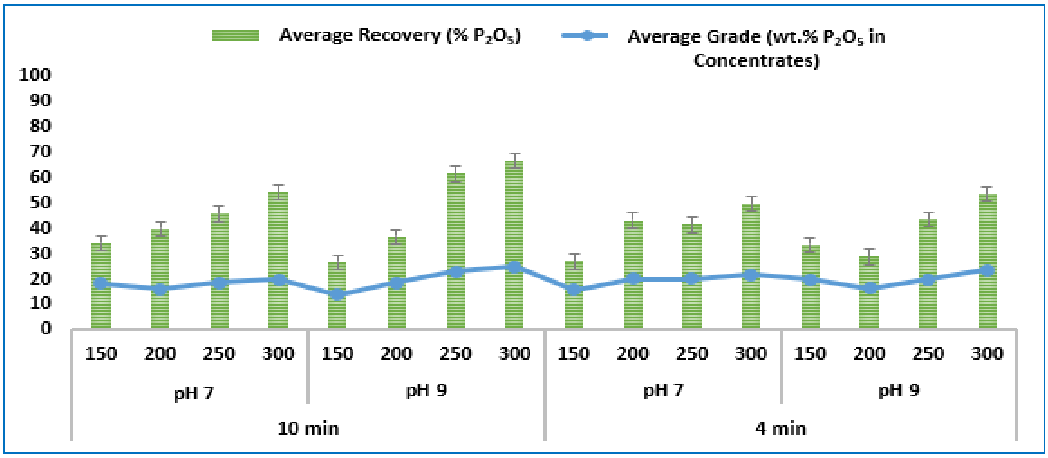

Fig. 7. Flotation efficiency of phosphate minerals in the presence of chitosan at different conditions

were determined to be 85/28.4%, 73/22%, and 46/19% with Hy-PAM, sodium silicates, and chitosan, respectively. When pH increased to 9 while keeping the dosage and the time at 250 g/ton and 10 min, the %recoveries/grades were determined to be 72/28%, 55/25%, and 62/23% with Hy-PAM, sodium silicates, and chitosan, respectively.

3.2.4. Summary and discussion of results from flotation tests

Results obtained from the flotation tests with chitosan and Hy-PAM indicated that both polymers can be used as potential depressants and outperformed sodium silicates depressant under specific conditions. However, Hy-PAM seems to be more flexible and can work effectively over a wider range of flotation conditions. As indicated by zeta potential measurements and supported by flotation tests, both Hy-PAM and chitosan showed preferential adsorption on quartz surfaces compared to apatite which resulted in silicate's depression to different extents. In the case of Hy-PAM polymer, the positively charged $Al(OH)_3$ cores are anticipated to adsorb on the surface of negatively charged quartz particles probably via electrostatic attraction mechanism which resulted in charge neutralization that was probably followed by bridging flocculation (Alagha et al., 2011; Alsafasfeh, 2020). This interaction should inhibit the attachment of quartz particles to air bubbles and thus suppress their flotation (Alagha et al., 2011; Alsafasfeh et al., 2018; Ontlametse Molatlhegi & Alagha, 2016). Overall, the selectivity of Hy-PAM adsorption on silicates could be enhanced at lower pH values as presented in Fig. 4. As shown, the interaction of Hy-PAM with apatite is minimal at pH 7 compared to pH 9 as indicated from the magnitude of the change in zeta potential ($\Delta\zeta$) of apatite after the addition of Hy-PAM at both pH values. In the case of quartz, there was a significant shift of zeta potential values of quartz suspensions after mixing with Hy-PAM at both pH values. For chitosan polymer, chitosan molecules possess positive charge which is expected to promote its adsorption on quartz. As shown in **Fig. 4**, the selectivity of chitosan's adsorption on quartz increased at higher pH (pH = 9). A study by Feng et al., revealed that at higher pH values, and in addition to the electrostatic interaction between chitosan and quartz surface, the decrease in chitosan's solubility would result in its deposition on the surface of quartz and increased the adsorption density of the polymer on quartz surfaces. This increase in the amount of polymer adsorbed may inhibit collector adsorption and lead to flotation depression (Feng et al., 2017; Schatz et al., 2003; Tiraferri et al., 2014). This may explain why higher pH gave better flotation efficiency of phosphates from silicates when chitosan was utilized. In general, these results were comparable to results from previous studies (Miller, 2001; Zhang, 2013) wherein polymers were sucessfully used as potential depressants in phosphate flotation under specific conditions.

3.3. Artificial neural network

The experimental dataset obtained from the flotation tests were used to train and test the performance of the ANN model (Table S2, SI). As presented in Fig. 8, four process variables were used as model input data: depressant type as a categorical variable (sodium silicate, Hy-PAM, and chitosan), depressant dosage (150, 200, 250, 300 g/ton), pH (7 and 9), and flotation time (4 min and 10 min) as numerical features. Both recovery and grade of P_2O_5 were used as model output data. The categorical variable (depressant type) was converted into numerical data using One-Hot-Encoder (Ul Haq et al., 2019). Data normalization was utilized as it is considered as the best practice for training the ANN to obtain a mean close to 0, which would speed up the learning process and lead to faster convergence between the dataset (Singh & Singh, 2020).

The number of hidden layers of the ANN model was varied between one, two, and three hidden layers. Then a comparison between real values and predicted values of the training and testing data was plotted to choose the best suitable hidden layer for the prediction and optimization process. Three main indicators (Mean Absolute Error "MAE", Coefficient of Determination "R^2", and Root mean square error "RSME") were used to evaluate the performance of ANN model. As shown in Fig. 9 and Table 2, the best model performance (in terms of recovery and grade) was obtained when three-hidden layers were used. Three-hidden layers gave the highest value of R^2 and lowest values of MAE and RMSE for both recovery and grade of phosphate minerals (presented as % P_2O_5). The calculated MAE, R^2, and RMSE for phosphate recoveries were 2.1%, 98%, and 4.0%, respectively. For phosphate grades, the calculated MAE, R^2, and RMSE were 0.32%, 99%, and 0.42%. Fig. 10 (A and B) shows good consistency between the experimental (real) training data and the predicted data for both of recovery and grade of P_2O_5. The results indicated that the optimum flotation performance was obtained in #10 and #40 at 10 min. This corresponds to Hy-PAM's dosages of 250 and 300 g/ton at pH 7 and 9, respectively.

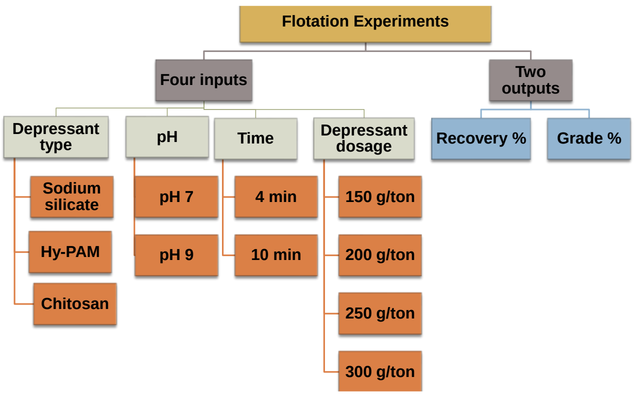

Fig. 8. Datasets developed from laboratory scale batch flotation experiments of silicate-rich phosphate tailing.

Table 2. Performance evaluation of the developed ANN model

ANN hidden layer	Flotation performance	MAE		R^2		RMSE	
		Training	Testing	Training	Testing	Training	Testing
One-hidden layer	Recovery:	12.9%	15.96%	12.49%	7.14%	15.99%	19.58%
	Grade:	3.46%	3.19%	25.62%	24.53%	4.17%	3.61%
Two-hidden layer	Recovery:	4.79%	4.30%	84.16%	93.88%	6.84%	5.02%
	Grade:	1.53%	1.20%	59.74%	85.01%	2.36%	1.6%
Three-hidden layer	Recovery:	2.14%	2.83%	97.83%	96.05%	2.51%	4.03%
	Grade:	0.32%	0.75%	98.72%	86.67%	0.42%	1.51%

3.3.1. Process optimization

One hundred random inputs were generated to optimize the flotation conditions for each depressant type (i.e., sodium silicate, Hy-PAM, and chitosan) that includes: depressant dosage which was varied from 150 g/ton-to-300 g/ton, flotation time which was varied from 4 min-to-10 min, and pH which was varied from 7-to-9. The optimum values of these parameters were selected based on the highest predicted flotation outcomes (%recovery and %grade of P_2O_5). Fig. 11 shows the predicted recovery and grade of P_2O_5 at a hundred random flotation conditions in the presence of sodium silicate, Hy-PAM, and chitosan, respectively. The base recovery and grade of P_2O_5 obtained from baseline experiments (experiments conducted using collector and frother only without the addition of any depressant as described in section 2.4) were used as the minimum values for the optimization process and are lined in red. All predicted values above the red line indicated a good flotation performance. In the presence of sodium silicate dispersant, higher flotation efficiency in terms of recovery and grade was predicted at longer flotation time >7 min, higher dispersant dosage (> 250g/ton), and medium pH range (pH 7 – pH8). The results in Fig. 11 indicated that the optimum flotation conditions in the presence of Hy-PAM were at shorter flotation time (time ~ 4 min), higher polymer dosage (dosage > 250g/ton), and lower pH range (pH 7 – pH8). However, optimum flotation efficacy of phosphates in the presence of chitosan can be obtained at flotation time between 5-7 min, high pH between pH 8 and pH 9, and higher chitosan dosage (>250 g/ton).

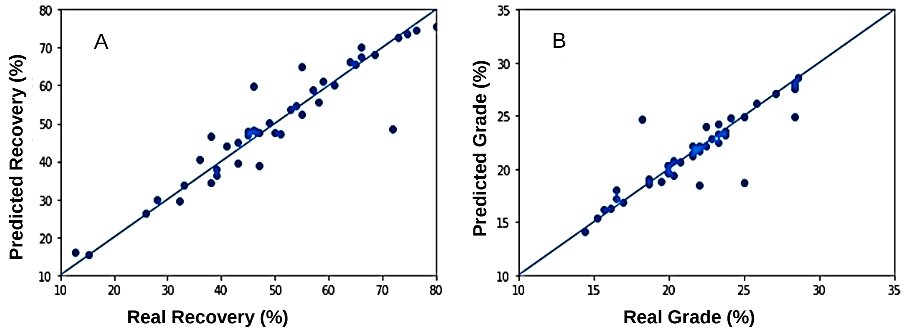

Fig. 9. Real (experimental) vs. predicted values of (A) the recoveries and (B) the grades of phosphate minerals (presented as % P_2O_5) in the training at three-hidden layer training phase.

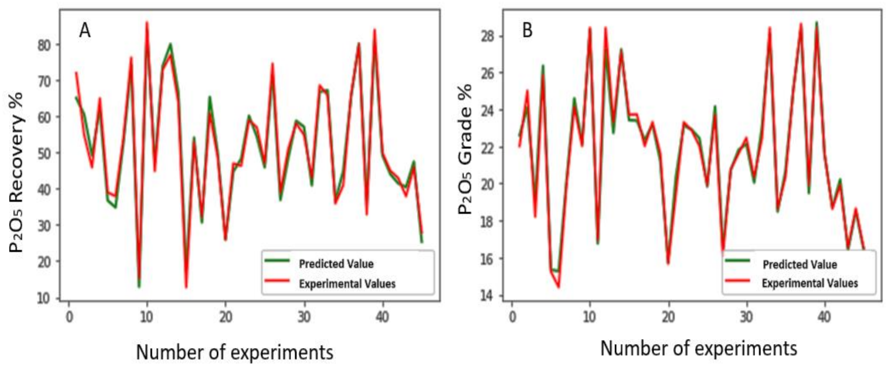

Fig. 10. Predicted vs. experimental data of training data for (A) the recovery and (B) the grade of phosphate minerals (presented as %P_2O_5)

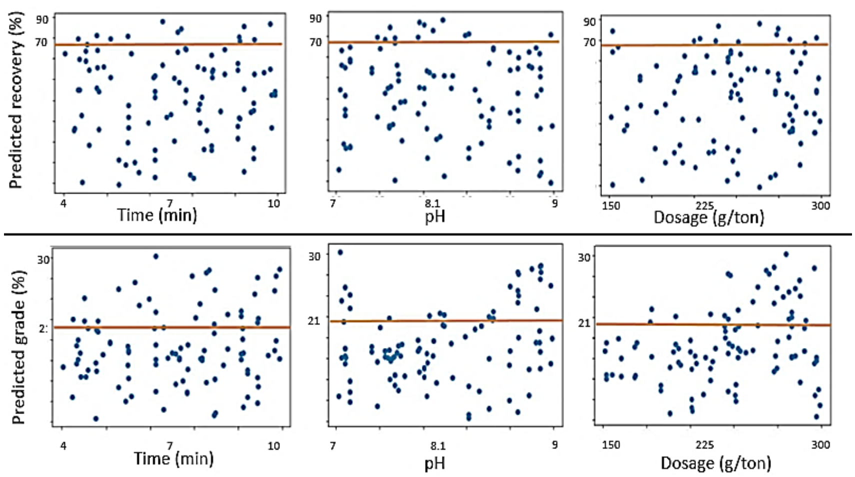

Fig. 11. Predicted recovery and grade vs. random flotation conditions in the presence of Hy-PAM (proposed silicate's depressant)

4. Conclusions

This study focused on improving flotation efficiency of phosphate minerals from silicate-rich phosphate mine tailings by utilizing biocompatible and selective polymers. In-house synthesized hybrid polyacrylamide-based polymer (Hy-PAM) and natural biodegradable polymers (chitosan) were tested as alternatives to conventional inorganic (i.e., sodium silicates) depressants of silicate minerals in the direct flotation of phosphate tailing samples. Zeta potential measurements showed a preferential adsorption of Hy-PAM and chitosan on quartz/silicates compared to apatite which makes both polymer good potential candidates to depress the flotation of silicates and thus enhance the flotation efficacy of phosphate minerals. Froth flotation tests of phosphate tailing samples were conducted in the presence of sodium silicate, Hy-PAM, and chitosan at different flotation conditions, including depressant dosages, pH, and flotation time. Overall, the optimum flotation efficiency of phosphate minerals (peak recovery and grade) was obtained when Hy-PAM was utilized at a dosage of 250 and 300 g/ton at pH 7 and 9, respectively, and flotation time of either 4 or 10 min. Results obtained from the flotation tests with chitosan and Hy-PAM indicated that both polymers can be used as potential depressants and outperform sodium silicates depressant under specific conditions. However, Hy-PAM seems to be more flexible and can work effectively over a wider range of flotation conditions.

All datasets produced from the flotation experiments were assimilated using the artificial neural networks (ANNs) model. The training and validation process of the ANN platform was employed to predict the flotation outcomes (recovery and grade) in relation to variables related to pulp and reagents characteristics (flotation time, pH, depressant dosage, and depressant type). Results obtained from the developed ANN model were used to optimize the flotation performance of phosphate minerals. Three main indicators (Mean Absolute Error "MAE", Coefficient of Determination " R^2", and Root mean square error "RSME") were used to evaluate the performance of the developed ANN model. The results showed that the highest value of R^2 and lowest value of RMSE for both recovery and grade (97.83%, 4.03%, and 98.72%, 0.42%, respectively) were obtained when three-hidden layers were used. Optimization results showed that the optimum flotation performance in the presence of Hy-PAM for both recovery and grade could be obtained at high dosage, low pH, and short flotation time. Moreover, in the presence of chitosan, the optimum flotation performance could be achieved at higher dosages, medium pH, and longer flotation time.

This research involves new ideas toward introducing novel biocompatible and functional reagents as process aids in the flotation of phosphate ores to further improve the sustainability of the process specially when recovering phosphates from secondary resources such as plant tailings. Moreover, the work presented here contributes immensely to the ongoing efforts for improving the performance predictability to enable better control of flotation systems to ensure process stability and peak performance.

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Supplementary Information (SI)

SI.1. Mineral characterization

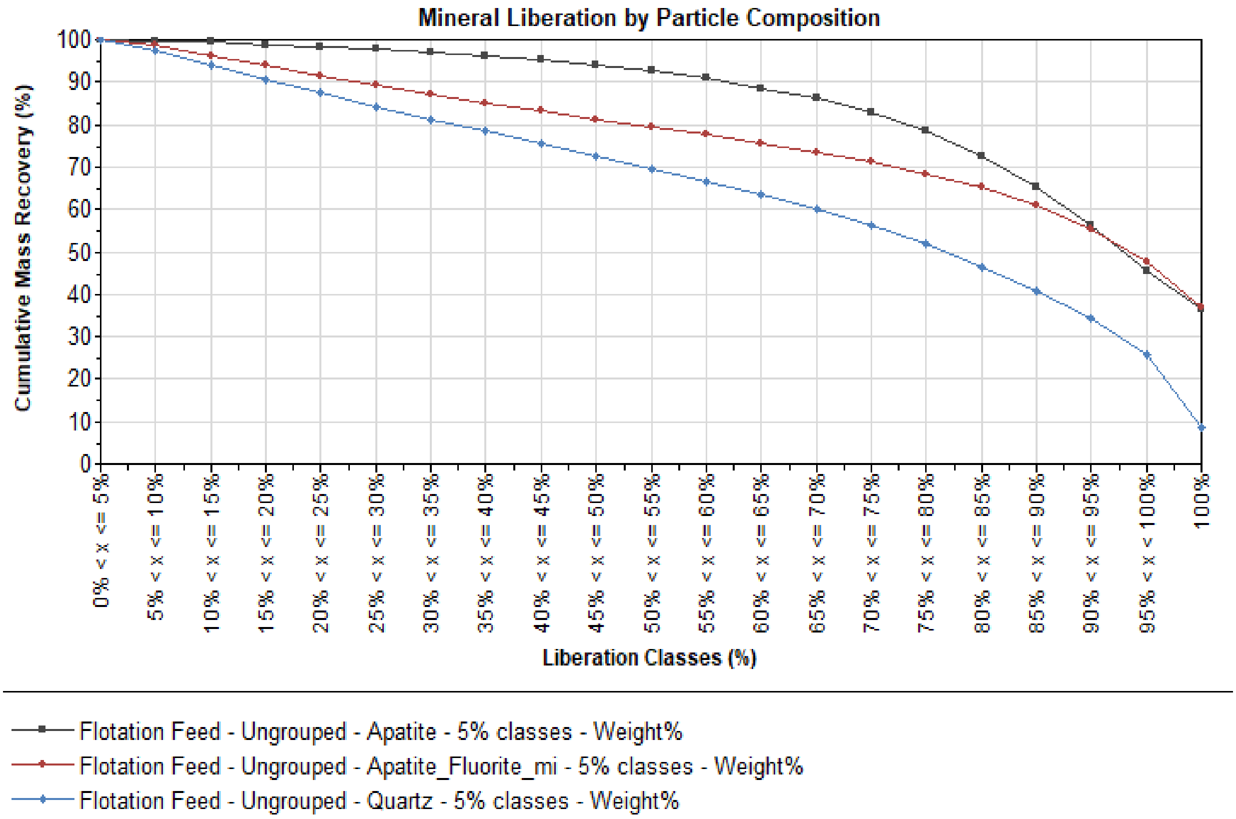

Fig. S1. Minerals liberation by particle composition for the apatite, mixed apatite/fluorite, and quartz

Table S1. SEM/EDS analysis of flotation feed

Element	Wt.%	At%	Element	Wt.%	At%
O	30.66	48.73	**Cl**	0.15	0.1
F	10.03	12.4	**Cd**	0.42	0.09
Na	4.85	4.95	**K**	1.14	0.68
Mg	1.43	1.38	**Ca**	18.73	10.98
As	0.36	0.11	**Ti**	0.2	0.1
Al	3.29	2.86	**V**	0.34	0.16
Si	11.53	8.24	**Cr**	0.36	0.16
P	7.63	5.16	**Fe**	1.9	0.8
Hg	0	0	**Zn**	5.37	1.93
S	1.61	1.18	**Total**	100	100

SI.2 Zeta potential measurements

Samples were prepared at 0.1 wt % of mineral in a 0.1 M KCl background solution. The prepared mineral suspensions were agitated using an IKA RW20 mechanical stirrer (IKA Instruments, Wilmington, NC, USA) for 45 min at a constant agitation rate of 250 rpm. The suspensions were allowed to settle for overnight. The supernatant liquid was considered for all zeta potential measurements. In all experiments, the solution pH was adjusted using either 1 M HCl or 1 M NaOH as needed.

SI.3. Flotation procedures and datasets

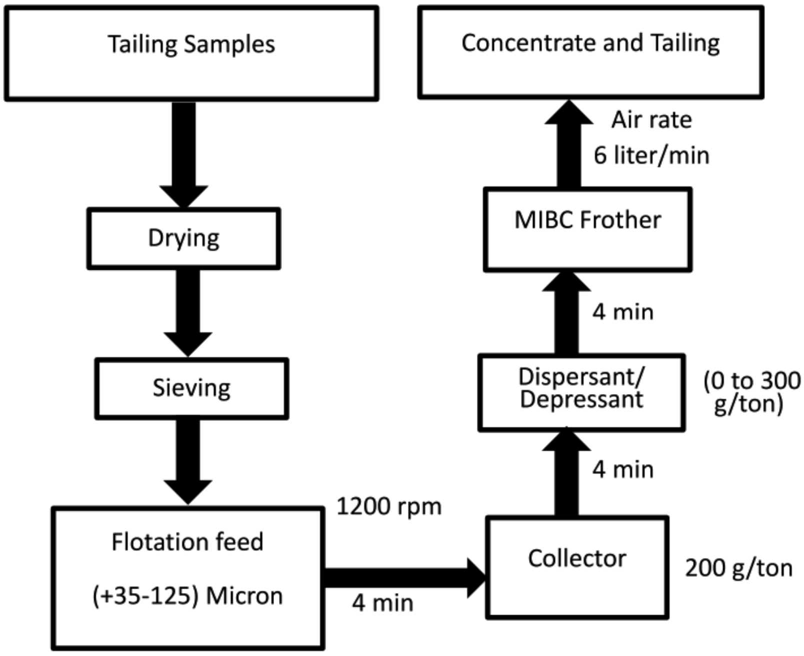

Fig. S2. A flowchart of the flotation experimental procedures used in this study

Table S2. Experimental datasets used in developing the ANN model for the flotation process of phosphate tailings

Experiment #	Depressant type	Flotation time	Pulp's pH	Depressant dosage (g/ton)	Solid concentration wt. %	%Recovery P_2O_5	%Grade P_2O_5
1.	No depressant	10 min	pH9	0	20%	32.27	22.03
2.	No depressant	10 min	pH9	0	40%	68.61	22.46
3.	No depressant	10 min	pH9	0	60%	77.06	23.31
4.	No depressant	4 min	pH7	0	20%	15.22	22.03
5.	No depressant	4 min	pH7	0	40%	24.76	21.61
6.	No depressant	4 min	pH7	0	60%	58.16	21.61
7.	No depressant	10 min	pH7	0	20%	10.34	21.61
8.	No depressant	10 min	pH7	0	40%	18.20	22.88
9.	No depressant	10 min	pH7	0	60%	76.33	24.15
10.	No depressant	4 min	pH9	0	20%	12.82	23.73
11.	No depressant	4 min	pH9	0	40%	46.43	23.31
12.	No depressant	4 min	pH9	0	60%	74.61	23.73
13.	Sodium silicate	10 min	pH7	250	60%	72.70	22.03
14.	Sodium silicate	10 min	pH7	200	60%	45.09	16.95
15.	Sodium silicate	10 min	pH7	150	60%	46.23	18.64
16.	Sodium silicate	4 min	pH7	200	60%	59.35	22.88
17.	Sodium silicate	10 min	pH9	150	60%	57.22	22.03
18.	Sodium silicate	10 min	pH9	250	60%	55.40	25.00
19.	Sodium silicate	4 min	pH9	250	60%	73.85	28.39
20.	Sodium silicate	10 min	pH7	300	60%	61.85	23.31

21.	Sodium silicate	4 min	pH9	300	60%	39.04	15.25
22.	Sodium silicate	4 min	pH7	150	60%	66.03	28.39
23.	Sodium silicate	4 min	pH9	200	60%	46.09	18.22
24.	Sodium silicate	10 min	pH9	200	60%	57.38	21.19
25.	Sodium silicate	10 min	pH9	300	60%	38.84	14.41
26.	Sodium silicate	4 min	pH7	300	60%	58.34	23.73
27.	Sodium silicate	4 min	pH9	150	60%	51.04	20.76
28.	Sodium silicate	4 min	pH7	250	60%	70.13	22.03
29.	Hy-PAM	10 min	pH7	150	60%	47.19	19.92
30.	Hy-PAM	4 min	pH9	150	60%	50.62	21.61
31.	Hy-PAM	4 min	pH9	300	60%	75.76	28.39
32.	Hy-PAM	10 min	pH7	250	60%	84.79	28.39
33.	Hy-PAM	4 min	pH7	150	60%	47.09	19.49
34.	Hy-PAM	10 min	pH9	300	60%	86.83	28.39
35.	Hy-PAM	4 min	pH7	250	60%	80.58	28.60
36.	Hy-PAM	10 min	pH9	200	60%	71.90	30.08
37.	Hy-PAM	10 min	pH9	150	60%	46.86	21.19
38.	Hy-PAM	4 min	pH7	200	60%	55.33	22.46
39.	Hy-PAM	10 min	pH7	200	60%	44.59	18.64
40.	Hy-PAM	10 min	pH9	250	60%	71.89	27.97
41.	Hy-PAM	10 min	pH7	300	60%	38.73	16.53
42.	Hy-PAM	4 min	pH9	250	60%	64.63	27.12
43.	Hy-PAM	4 min	pH9	200	60%	65.43	25.85
44.	Hy-PAM	4 min	pH7	300	60%	42.96	17.37
45.	chitosan	4 min	pH7	250	60%	41.46	20.34
46.	chitosan	10 min	pH9	300	60%	66.72	25.00
47.	chitosan	4 min	pH9	250	60%	43.43	19.92
48.	chitosan	4 min	pH7	300	60%	49.76	21.61
49.	chitosan	10 min	pH7	200	60%	39.86	16.10
50.	chitosan	4 min	pH9	300	60%	53.58	23.73
51.	chitosan	10 min	pH7	250	60%	45.93	18.64
52.	chitosan	10 min	pH9	250	60%	61.60	22.88
53.	chitosan	10 min	pH7	150	60%	34.29	18.22
54.	chitosan	4 min	pH7	150	60%	26.99	15.68
55.	chitosan	4 min	pH9	150	60%	33.63	19.92
56.	chitosan	10 min	pH9	150	60%	26.70	13.98
57.	chitosan	10 min	pH7	300	60%	54.20	19.92
58.	chitosan	4 min	pH9	200	60%	28.92	16.53
59.	chitosan	4 min	pH7	200	60%	43.12	20.34
60.	chitosan	10 min	pH9	200	60%	36.80	18.64