

STUDY OF IN-FILTER PHOSPHORUS REMOVAL MECHANISMS IN AN AERATED BLAST FURNACE SLAG

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Abstract

The removal mechanism of phosphorus in a wastewater treatment system remained uncertain due to their process complexity. Accordingly this study has been conducted to investigate the removal mechanism of phosphorus from domestic wastewater in an aerated blast furnace slag filter. In this study, several blast furnace slag (BFS) samples (fresh, after 2,4 and 6 months treatment period) and particulates (4-5 mm in size) and sediments samples from the BFS filter after one year operation on site from the pilot-scale aerated BFS filter were taken for scanning electron microscopy (SEM) examinations and X-ray diffraction (XRD) analysis to study the changes of the filter media after usage in the ARF system. Scanning electron microscopy and X-ray diffraction spectrometric analyses on the surface of BFS, particulates and sediment samples revealed that the apparent mechanisms of P-removal in the filter are adsorption on the amorphous oxide phase of the BFS surface and precipitation within the filter.

Index Terms: Blast furnace slag, domestic wastewater, phosphorus, removal mechanism,

1. INTRODUCTION

Phosphorus removal mechanisms in a rock filter RF system are very similar to the removal in a constructed wetland (CW) system with the exception of the plant uptake in a CW treatment. From previous studies carried out on CW systems, it is clearly stated that the main phosphorus removal occurs by adsorption and retention by substrates [1], plant uptake [2], microbial immobilization [3], and precipitation in the water column [4,5] as well as adsorption, complexation and precipitation with aluminium (Al), iron (Fe), calcium (Ca) and clay minerals in the bed matrix [6]. Metcalf and Eddy [7] also indicated that, in the case of natural treatment systems, the major removal mechanisms of phosphorus are through adsorption and chemical precipitation. The phosphorus, which is mainly in the forms of orthophosphates, was reported to be absorbed by clay minerals and various organic soils fraction in the soil matrix and chemically precipitate with Ca (at neutral to alkaline pH values) and iron or aluminium (at acid pH values) [7].

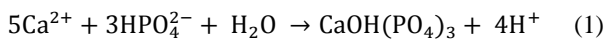
In a subsurface-flow system –which is identical to the ARF systems used in the present study where wastewater is in direct contact with the adsorbents – adsorption and retention is the main pathway of phosphorus removal [8]. Moreover, Crites et al. [9] point out that the main pathways for subsurface CW are adsorption and precipitation, and Bubba et al., [10] point out that the adsorption and precipitation of phosphorus in CW is a very complex phenomenon which can

occur simultaneously. The filter media can both adsorb the phosphate ions and/or promote its precipitation by supplying the solution with metals, which can react with phosphorus to produce sparingly soluble phosphorus.

Under aerobic conditions, such as in an ARF system, insoluble phosphates are chemically precipitated with ferric iron; Ca and Al tend to be adsorbed onto adsorbent surfaces, such as clay particles, ferric and Al hydroxides and oxides [8]. This condition is identical to that in aeration tanks in an activated sludge system. Gerardi [11] indicates that, when orthophosphate, polyphosphate and organic phosphorus compounds enter an activated sludge process, they subsequently undergo biological and chemical changes and thereby experience several fates. Furthermore, various organic phosphorus compounds are removed from the wastewater when particulate organic phosphorus is absorbed by solids and settled out. The compounds are degraded through microbial decomposition with the principal organisms activities including actinomycetes, such as Streptomyces; bacteria, such as Arthrobacter and Bacillus, and fungi, such as Aspergillus and Penicillium [11]. These organisms produce an enzyme called phosphatase which releases orthophosphate from phosphorus-containing organic compounds.

Furthermore, under aerobic conditions, some polyphosphate is hydrolyzed to orthophosphate, which is a readily available nutrient compound for bacterial growth and energy transfer. Notably, some organic phosphorus is decomposed to release

phosphorus. Gerardi [11] also stated that orthophosphate in an aeration tank may be incorporated into floc particles as soluble hydroxyapatite ($\text{CaOH}(\text{PO}_4)_3$). In aerobic conditions where dissolved oxygen is relatively high and much carbon dioxide is stripped to the atmosphere, little carbonic acid is produced; this will then increase the pH levels in the system. Under this condition, orthophosphate is released and present as the HPO_4^{2-} ion. With the system containing Ca, orthophosphate is precipitated from the solution as hydroxyapatite and subsequently incorporated into floc particles. The naturally occurring reaction is in equation (1):



After the processes have been completed, the final effluent will leave the treatment system with approximately 90% orthophosphate.

To date, phosphorus-removal mechanisms involved in an RF system remain uncertain due to the complexity of phosphorus during the overall removal process, despite the fact that the subject has now been studied for more than 20 years [12]. Nevertheless, it remains evident that more detailed research is required in order to elucidate the sorption of phosphate [13]. Furthermore, Drizo et al. [14] have highlighted that the actual removal mechanisms of phosphorus by slag filter 'remain uncertain and should be investigated in future studies'. Notably, although there have been a few preliminary investigations on this subject, there has been no comprehensive study to take into account the various possible phosphorus-removal mechanisms by slag filters, either with the use of electric arc furnace slag (EAFS) or BFS as the filter media. Pratt et al. [15], however, point out that, to date, phosphorus-removal mechanisms of those filter media used to treat effluent are also poorly understood; this is owing to the fact that the main pathway of phosphorus-removal in a slag filter is regulated by pH, temperature and concentrations of coexisting metallic salts within the treatment system, as has been discussed in Section 2.5.2.3. The main apparent phosphorus-removal pathway in the BFS and other slag filters are adsorption on to the slag surface and precipitation of metallic phosphate salts. Previous laboratory investigations (e.g., [12, 16, 17]) utilizing BFS concluded that the dominant removal mechanism at $\text{pH} < 4.5$ and $\text{pH} > 8.5$ was precipitation and adsorption respectively, as shown in Figure 8.3. Furthermore, under neutral pH to alkaline conditions ($7 < \text{pH} < 14$), the adsorption of phosphate to calcium occurs and tricalcium phosphate is produced by the reaction between calcium and dihydrogen phosphate, as shown by Brady [18] in equation (2):



Baker et al. [19] indicate that metal oxides were important phosphate (PO_4^{3-}) adsorption sites owing to multiple charged

cautions, high positive surface charge densities at near-neutral pH, and a propensity to hydroxylate in the case of aqueous systems. Consequently, this study was designed in order to establish the possibility of a phosphorus-removal mechanism within the aerated BFS filter treating facultative pond effluent containing 1- 3 mg P/L at neutral to mildly alkaline pH (6.8-9.0).

2. MATERIALS AND METHODS

2.1 Blast Furnace Slag (BFS) Samples

BFS samples from the pilot-scale filter as shown in Figure 1 were taken for scanning electron microscopy (SEM)/EDX examinations and X-ray diffraction (XRD) analysis to study the changes of the filter media after usage in the subsurface horizontal-flow Blast-Furnace Slag Filter system. In the present work, samples of fresh BFS and BFS of the subsurface horizontal-flow Blast-Furnace Slag Filter from Esholt Wastewater Treatment Works, Bradford, UK treating PFP effluent, were collected after three periods of operation of 2, 4, and 6 months, and analyzed for SEM/EDX. Particulates (4-5 mm in size) and sediments samples from the BFS filter after one year operation on site also were taken for XRD analysis.



Figure1. The subsurface horizontal-flow Blast-Furnace Slag Filter (left); blast furnace slag (right)

2.2 Scanning Electron Microscopy (Sem) Analysis

Scanning electron microscopy (SEM) analyses were carried out to study the BFS surface using very high magnifications. BFS samples were placed in the oven immediately after collection from the experiment station to dry for 24 hours at 105°C [14] before they were allowed to cool in a desiccators. The BFS samples were then crushed to about 0.5 cm in size in a mortar and pestle. The crushed samples were soaked in acetone in an ultrasonic shaker for a few minutes to remove loose material on the surface. Later, the cleaned samples were dried on a hot plate at 110°C and mounted on the specimen holder with cold setting glue. Finally, the mounted samples were coated with a thin layer (2–3 minutes coating time) of

gold using an Emscope SC500 Gold Sputter Coater (Emscope, Ashford, UK) before further examination by SEM. The analyses have been conducted in the Leeds Electron Microscopy and Spectroscopy Centre Institute for Materials Research (LEMAS), School of Process Environmental and Materials Engineering, University of Leeds.

The prepared samples were then examined and photographed using an Environmental Scanning Microscope, XL30 ESEM (Philips, UK) to facilitate identification of the elemental composition of the samples. The ESEM is fitted with a secondary electron detector (SE), a back scatter electron detector (BSE) and a gaseous secondary electron detector (GSE). It also had an Oxford Instruments INCA ENERGY (EDX) x-ray analysis system. The SEM micrographs, EDX spectrum and elemental semi quantitative table will be produced of each sample. The information will be analyzed subsequently to study phosphorus present and distribution on the fresh and treated BFS surface samples.

2.3 X-Ray Diffraction (XRD) Analysis

X-ray diffraction (XRD) analysis is widely used for structural and phase composition studies. XRD is particularly useful for mineralogy structure identification and also identification of the chemical elements present in the geological samples. In the present study, fresh and after 6 month treated BFS, particulates (4-5 mm) and sediment samples from the BFS filter after one year operation on site at Esholt WWTP, were placed in the oven immediately after collection from the experiment station to dry for 24 hours at 105°C [14] before they were allowed to cool in a desiccators. Later the samples were processed for X-ray diffraction (XRD) with an X-ray diffractometer equipped with a copper tube (Cu K α) radiation and a graphite monochromator (model PW1050-Cu K α , Royal Philips Electronics N.V., Amsterdam) in the School of Earth and Environment, University of Leeds. Acquisition was performed on a spinner sample stage running in continuous scan mode over the range from 5.000° to 70.005° 2 θ with a step size of 0.010 giving a total of 31 steps. The analysis method was based on a comparison of the values of interplanar distances and intensities of peaks in diffractograms of samples and a reference sample, using the standard database of ICDD PDF-2 of the International Centre for Diffraction Data. After XRD analyses, spectrum pattern produced of each sample will be analyzed to study the changes of peak produced between fresh and treated BFS surface samples. As for the sediment samples, minerals contained in the samples will be characterized through the peak produced on the spectrum.

3. RESULTS & DISCUSSION

Spectroscopic analyses—including SEM/EDX and XRD—were used in the present study in order to investigate phosphorus distribution on the BFS surface and in sediment samples. Fresh and treated BFS were characterized using both

SEM/EDX and XRD, whilst particulate (4-5 mm) and sediment samples from the BFS filter were examined with the use of XRD. Results from both analyses for both fresh and treated BFS, particulates and sediment samples are discussed in the following sections.

3.1 Chemistry and Mineralogy of Fresh BFS

The mineral and chemical composition of fresh BFS samples from Tarmac Quarry Products Ltd., Scunthorpe, UK, was characterized through spectroscopy examinations. In terms of its mineralogy, as been discovered through XRD analysis, fresh BFS is considered to be a crystalline glassy mineral mainly consisting of melilite ($\text{Ca}_4\text{Al}_2\text{MgSi}_3\text{O}_{14}$ / $4\text{CaOAl}_2\text{O}_3\text{MgO}\cdot 3\text{SiO}_2$) and gehlenite: $\text{Ca}_2\text{Al}_2\text{SiO}_7$. In addition, X-ray Fluorescence (XRF) was used in order to determine the chemical composition of the BFS. The results of the XRF analyses suggest that the elemental chemistry is dominated CaO (39.37%), SiO₂ (35.29%), Al₂O₃ (12.65%) and MgO (8.69%). Moreover, the low iron content (<1% by weight) and the low carbon content (<1% by weight) were determined by the loss on ignition (LOI) test, which suggests that adsorption and precipitation processes in the BFS filter system are controlled by the interactions with reactive calcium, silica, aluminium and magnesium sites, and not via interactions involving iron oxides.

Furthermore, as has been gathered from the SEM/EDX analysis, the most abundant minerals on a fresh BFS surface were found to be calcium and silica, as shown in the SEM photomicrographs and EDX spectra in Figure 2 which confirm that no phosphorus was found on the fresh BFS surface.

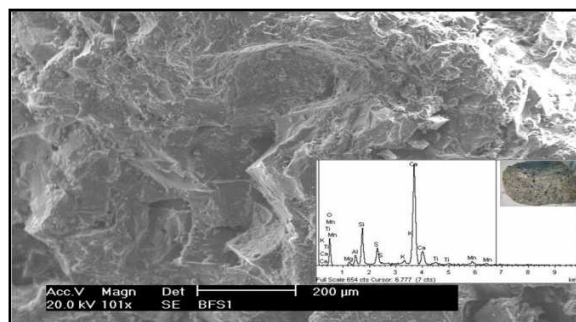


Figure2. SEM micrograph of surface scanned and EDX spectrum of Fresh BFS sample

3.2 BFS Samples after Treatment

After a few months in the BFS filter system, phosphorus can be seen on the surface of BFS samples after being examined by SEM/EDX. Figures 3(a) & (b) show the presence of phosphorus on the surface of BFS through EDX mapping and the spectrum analysis of surface BFS samples after a two-month period of the treatment. SEM micrographs and EDX spectra analyses four- and six-months after treatment in the

BFS filter are detailed in Figures 4 and 5, respectively. Phosphorus was found on the BFS surface samples of each sample, as the phosphorus peaks have been clearly seen and revealed by the presence of phosphorus peaks on the EDX spectra. From a semi-quantitative analysis produced after EDX mapping on all BFS samples, it can be concluded that the accumulation of phosphorus on the BFS surface has a linear relationship to the contact time of BFS with wastewater. This condition can be described by analyzing the percentage by the weight of phosphorus accumulating on the surface following 0-, 2-, 4- and 6-month periods of treatment. The concentration of phosphorus accumulating on the BFS surface over a two-month period was approximately 1.59 ± 0.68 % by weight. The adsorbed phosphorus on the BFS surface gradually increased over the 6-month period of the trial, as shown in Table 1. The percentage of phosphorus determined on the surface of BFS from semi-quantitative analysis in this study was higher than that established by Pratt et al. [20], which was approximately 3.37% by weight of phosphorus in their slag samples. These results indicate that as the time of contact between BFS and wastewater increased, more phosphorus was expected to be adsorbed onto the BFS surface.

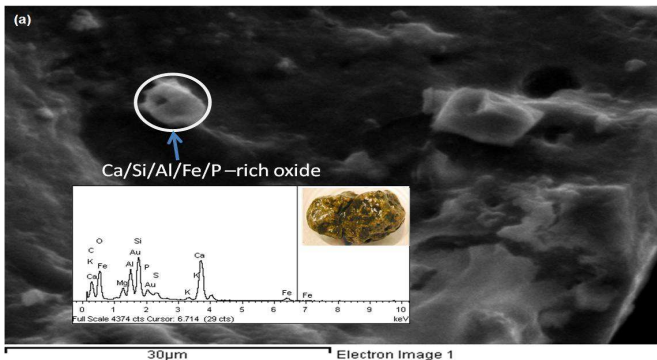


Figure 3(a). SEM micrographs and EDX spectra of BFS surface samples over a two-month treatment period in the aerated BFS filter

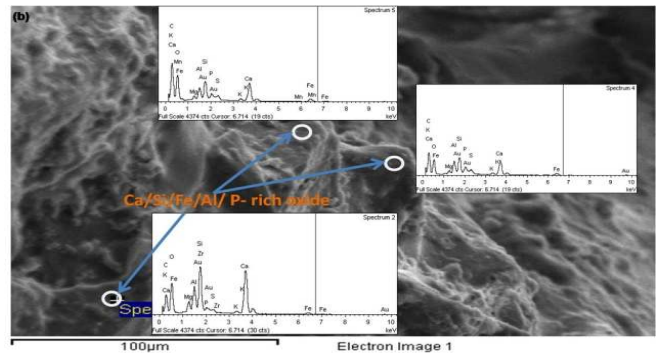


Figure 3(b). SEM micrographs and EDX spectra of BFS surface samples over a two-month treatment period in the aerated BFS filter

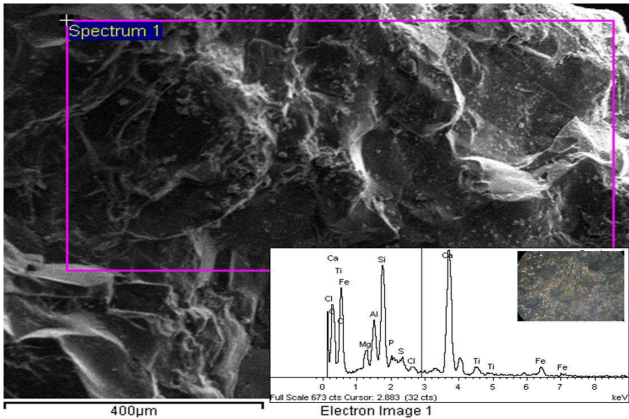


Figure4. SEM micrographs and EDX spectra of BFS surface sample over a four-month treatment period in the aerated BFS filter

Table 1 Elemental concentrations in Blast Furnace Slag surface sample as determined by EDXa

| BFS Samples | Elemental concentration (expressed in wt % of sample surface material) | | | | | | | |
|------------------|--|------|-----|------|------|------|-----|------|
| | P | Fe | Al | Ca | Mg | Si | S | C |
| Fresh | n.a. | n.a. | 4.9 | 18.5 | 3.9 | 12.6 | 0.8 | 0.9 |
| Treated (2-mths) | 1.6 | 2.2 | 3.6 | 6.4 | 1.1 | 4.7 | 1.4 | 41.6 |
| Treated (4-mths) | 2.2 | 4.9 | 2.9 | 22.3 | 0.6 | 6.08 | 1.7 | 31.4 |
| Treated (6-mths) | 6.5 | 23.9 | 0.4 | 7.5 | n.a. | 1.7 | 1.6 | 6.2 |

a Note not all element contents are shown therefore the percentage for each material do not add 100% (e.g. K, Au, Ti, Mn), n.a. not available

From the semi-quantitative analysis carried out on BFS samples, it can be concluded that phosphorus-rich oxides formed after the effluent from primary facultative pond underwent further treatment in the BFS filter. These findings demonstrate that BFS has a high capacity for adsorbing phosphorus from wastewater, as well as chemisorptions, which took place on the BFS surface. The BFS used in this study was comprised of 39% CaO and 35% SiO₂, as provided earlier in Table 1. Therefore, the adsorption of phosphorus to calcium oxide could have been the key removal mechanism

for phosphorus in the BFS filter. The results obtained from the BFS filter in the present study has a good agreement, as has been revealed by Pratt et al. [20] in the electric furnace slag filter treating WSP effluent at near-neutral pH value. They subsequently found that the key removal of phosphorus in the near neutral pH system was through adsorption onto Fe oxides/oxyhydroxides and oxidising Eh. Furthermore, de-Bashan and Bashan [21] also indicate that the main pathway of phosphorus-removal was achieved via adsorption onto hydroxide surface.

From the XRD scans of BFS samples after a six-month period of treatment on the surface of BFS samples, the spectrum did not show any significant changes in the mineralogy, which was mostly melilite ($\text{Ca}_4\text{Al}_2\text{MgSi}_3\text{O}_{14}/4\text{CaOAl}_2\text{O}_3\text{MgO} \cdot 3\text{SiO}_2$) and gehlenite ($\text{Ca}_2\text{Al}_2\text{SiO}_7$), with the exception of calcite and quartz peaks, as can be seen on the BFS surface following treatment in the filter (Figures 6 and 7). The present findings indicate that the accumulated and adsorbed phosphorus species, as shown in the previously mentioned SEM micrographs, were amorphous oxides.

Notably, amorphous oxides have higher phosphorus adsorption capacities than crystalline oxides due to their larger number of singly-coordinated surface hydroxyl ions, as has been reported by Vymazal et al. [22]. Therefore, as can be determined from the spectroscopy analyses on the BFS surfaces characterisation study, phosphorus adsorption occurs on amorphous Ca/Si/Al/Fe oxides in the BFS filter.

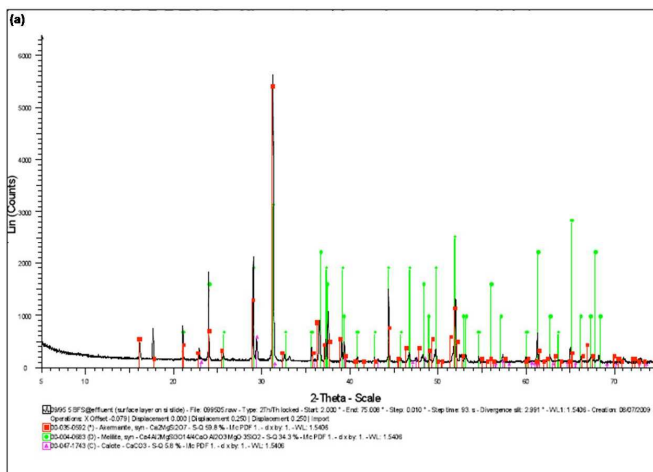


Figure6. X-ray diffraction diagram of the of BFS surface samples over a 6-month treatment period in the aerated BFS filter

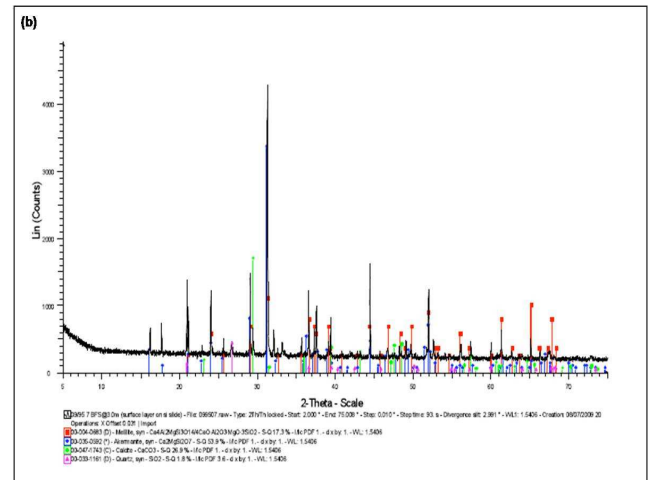


Figure7. X-ray diffraction diagram of the of BFS surface samples over a 6-month treatment period in the aerated BFS filter

3.3 Particulates (4-5 Mm) and Sediments Samples

Particulates within the range 4-5 mm and sediments were sampled from the BFS filter after one year's operation on-site at Esholt WWTP for mineral characterisation using X-ray diffraction. From the XRD analysis of particulates samples, it can be seen that particulates which were amorphous in the BFS filter consist of hydrated yttrium erbium phosphate, which are also known as churchite ($\text{YPO}_4 \cdot 2\text{H}_2\text{O}$) and potassium-rich halite ($\text{K}_0.2\text{Na}_{0.8}\text{Cl}$), as shown in Figure. The presence of phosphate-bearing minerals in the amorphous particulates samples shows that some phosphate ions react with inorganic compounds in the wastewater and precipitate as sediments in the bottom of the filter. This phenomenon was shown by the XRD analysis of the sediment samples. The phosphate-bearing mineral churchite ($\text{YPO}_4 \cdot 2\text{H}_2\text{O}$) was also present in sediment samples, as can be seen in Figure 8. From the particulates material and sediment sample characterisation study, which was carried out by XRD analysis, it can be concluded that precipitation has taken place in the BFS filter. Furthermore, Johansson and Gustaffson [23] also propose that the main mechanism of phosphorus removal in the BFS filter was through calcium phosphate, namely hydroxyapatite [$\text{HAP: Ca}_5(\text{PO}_4)_3\text{OH}$], precipitation. Nevertheless, although the precipitation of hydroxyapatite may be expected in the case of sediment samples owing to Ca dissolution within the BFS filter, no HAP was found in the samples; this might be due to the amorphous sediment samples as clearly shown in Figure 9. Therefore, it was quite problematic to analyze the sample. In addition, Vohla et al. [24] also conclude that, in a system containing a high content of CaO, the main process of phosphorus removal was precipitation.

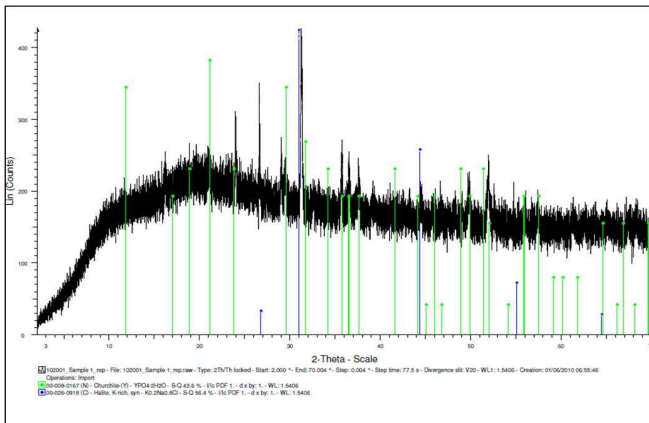


Figure8. X-ray diffraction scan of the particulate samples

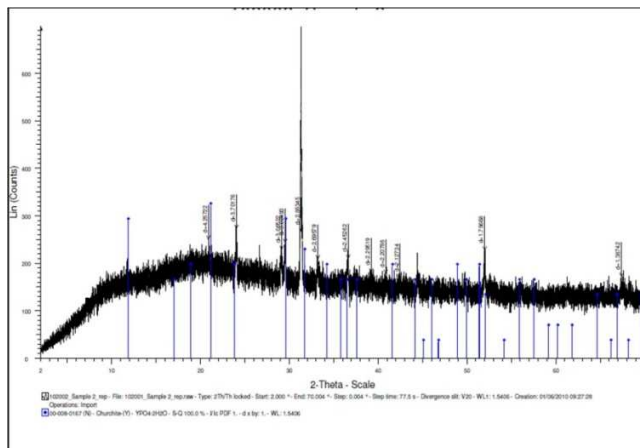


Figure9. X-ray diffraction scan of the of sediments samples

CONCLUSIONS

From the study concerning in-filter phosphorus removal mechanisms, the following conclusions have been drawn:

- The dominant mechanisms of phosphorus-removal in the filter treating facultative pond effluent involved the adsorption onto the BFS surface, as well as precipitation in a near neutral pH of the aerated BFS filter.
- Phosphorus from wastewater adsorbed onto amorphous Ca/Si/Al/Fe oxides of BFS surface.

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