

Enhanced Nutrients Removal in Conventional Anaerobic Digestion Processes

M. Z. Othman, S. Uludag-Demirer and G.N. Demirer

Abstract—One of the main challenges for one phase anaerobic digestion processes is the high concentration of NH_4^+ and PO_4^{3-} ions in the digested sludge supernatant. This project focuses on enhancing the removal of nutrients during the anaerobic digestion process through fixing both NH_4^+ and PO_4^{3-} ions in the form of struvite (magnesium ammonium phosphate, MAP, $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) within the anaerobic sludge. Batch anaerobic digestion tests showed that Mg^{2+} concentration in the range 279 – 812 mg/L had insignificant effect on CGP but incurred a slight increase in COD removal. The reactor that had soluble $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$ at a molar ratio of 1.28:1:00:1:00 achieved the best performance enhancement of 8% increase in COD removal and 32% reduction in NH_4^+ in the reactor supernatant. Overall, the results show that there is a potential to optimise conventional anaerobic digestion such that supernatant lean in P and N, and sludge rich in nutrients are obtained.

Keywords—Anaerobic Digestion, Nutrients, Struvite, Waste Activated Sludge (WAS)

I. INTRODUCTION

ONE of the main challenges of OPAD process is the high concentration of NH_4^+ and PO_4^{3-} ions in the digested sludge supernatant (or the filtrate from a sludge dewatering facility). The release of inorganic nitrogen (N) and Phosphorous (P) during anaerobic digestion as a result of biotransformation of proteins and solids has been reported by different researchers [1]-[3]. The low treatment efficiencies observed for N and total P are not surprising since anaerobic digesters are known to reduce negligible amounts of nutrients [4]. As a result, treatment plants employing anaerobic digestion also operate unit processes for the removal of N from the digester's effluent or return it back for further treatment upstream, secondary processes which add to the nitrogen loading onto the biological processes. Consequently increasing the cost associated with N and P removal.

Many studies investigated the potential removal and recovery of nutrients from anaerobic digestion effluents through formation of magnesium ammonium phosphate hexahydrate, ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$), commonly known as struvite [5]-[9]. The application of this approach requires the installation and operation of process units specifically for

struvite production, which adds to the wastewater treatment plant costs, including operation and maintenance. A study that looked at the feasibility of a full scale struvite recovery from anaerobic digestion effluents indicated the value of product, struvite, is only one third of the total annual cost of the process. Therefore another approach is to develop and optimise existing OPAD processes for enhanced nutrients removal. Limited research has been carried out in this field, where only two studies, of published literature accessible to the research team of this study, investigated the potential feasibility of the formation of struvite inside anaerobic digesters. The authors of [10] reported that struvite precipitation obtained by the addition of Mg^{2+} during anaerobic digestion led to 67% N and 73% P removal whereas [11] stated that the anaerobic reactors supplemented with struvite forming chemicals removed considerable amounts of NH_3 (10-23%) relative to the control reactor. The authors of [10] and [11] investigated the digestion of food waste and dairy manure respectively, which have different characteristics to sludge produced in domestic wastewater treatment plants. Therefore there is a need to research potential enhancement of conventional OPAD through struvite formation (i.e. nutrients fixing) for WAS generated at domestic wastewater treatment plants. This study aims to investigate the impact of the addition of the limiting constituents Mg^{2+} and PO_4^{3-} , for different combinations of Mg^{2+} only and combinations of Mg^{2+} and PO_4^{3-} , on the performance of the anaerobic digestion process in terms of biogas formation and recovery of NH_4^+ and PO_4^{3-} . An associated objective is to assess the effect of pH on the performance of the process.

II. MATERIALS AND METHODS

A. Materials

The sludge used is thickened waste activated sludge (WAS) obtained from a domestic wastewater treatment plant in Melbourne. The seed used was obtained from the anaerobic digester of another domestic wastewater treatment plant. Both the thickened WAS and seed samples were filtered through a 1.0 mm mesh, analyzed for the parameters shown on Table 1 and stored at 4°C prior to use. MgCl_2 and $\text{Na}_3\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ used are analar grade (Sigma Aldrich).

B. Experimental Set-up

Bench scale batch anaerobic digestion reactors of 120 mL size were used to assess the effectiveness of conventional anaerobic digestion with and without the addition of magnesium and phosphorous, to enhance removal of nitrogen

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and phosphorus generated during the digestion process. The biochemical methane production (BMP) potential experimental procedure outlined in [3] was followed.

The experimental set up comprised duplicate blank and control (represents conventional anaerobic digestion) reactors. Reactors R1 to R20 received varying dosages of MgCl₂ (technical grade, BDH chemicals) without or combined with varying dosages of Na₃PO₄·12H₂O (analytical grade, BDH chemicals) as the PO₄³⁻ source. Reactors NR1 to NR8 received the exact dosages added to R1 – R8 except that the initial pH was adjusted, pH = 8.2. The dosages and initial concentrations of Mg²⁺ and PO₄³⁻ are outlined in Table 2. Each reactor contained 25 mL of WAS and 25 mL of seed, except the blank which did not receive seed. After adding the chemicals, the reactor were sealed, mixed and put in a shaking water bath at 35±2C and 100 rpm. The reactors received varying dosages of Mg²⁺, to assess the potential of struvite formation, where NH₄⁺ and PO₄³⁻ are those naturally present and released during the process. Other reactors received both Mg²⁺ and PO₄³⁻ to enhance the potential of struvite formation, as they are usually both limiting in the AD reactor. The performance of the reactors was monitored by measuring the daily biogas production for 41 days.

C. Analytical

Chemical oxygen demand (COD), total P, P, PO₄³⁺, total N, NH₃-N, NH₄⁺, were measured using HACH Spectrophotometer (DR/4000). Mg²⁺ concentrations were measured using atomic absorption spectrophotometer (Varian SpectrAA-600). The gas produced in each reactor was measured using a water displacement device. The content of methane in the biogas was determined as follows. A known volume of the headspace gas (V1) produced in a serum bottle used in the biochemical methane production (BMP) experiments was syringed out and injected into another serum bottle which contained 20 g/L KOH solution. This serum bottle was shaken manually for 3-4 min so that all the CO₂ and H₂S were absorbed in the KOH solution. The volume of the remaining gas (V2), which was 99.9% CH₄, in the serum bottle was determined by means of a syringe. The ratio V2/V1 provided the content of CH₄ in the headspace. The methane content of biogas was determined in six duplicate samples and found to be 61.5±6.6.

TABLE I
CHARACTERISATION OF THE THICKENED WAS AND SEED SLUDGE
USED IN THIS STUDY

| Parameter (mg/L) | Thickened | Seed |
|--|-----------|--------|
| Total COD | 25,900 | 20,175 |
| COD Soluble | 4,680 | 3,010 |
| Total Solids | 12,098 | 15,635 |
| Volatile Solids | 10,000 | 10,515 |
| Total P | 957 | 2,345 |
| TP Soluble | 222 | 538 |
| [PO ₄ ³⁻] Soluble | 169 | 485 |
| Total N | 960 | 1,250 |
| Total N Soluble | 295 | 670 |

| | | |
|--------------------|------|-----|
| NH ₃ -N | 203 | 532 |
| Mg ²⁺ | 16.7 | 6.2 |
| pH | 5.7 | 8.0 |

TABLE II
THE DOSAGES ADDED TO, AND INITIAL CONCENTRATIONS OF
Mg²⁺, NH₄⁺, AND PO₄³⁻ IN, THE REACTORS OPERATED IN THIS
STUDY

| React- tors* | Chemicals Added (mg/L) | | Initial Concentration in the reactors (mg/L) | | |
|-----------------|------------------------------|----------------------------------|--|----------------------------------|----------------------|
| | Mg ²⁺ | [PO ₄ ³⁻] | Mg | [PO ₄ ³⁻] | NH ₄ + |
| Cont- rol | - | - | 11.8 | 411.5 | 427.5 |
| R1 | 266.8 | - | 278.6 | 411.5 | 427.5 |
| R3 | 266.8 | 912.2 | 278.6 | 1323.7 | 427.5 |
| R5 | 533.6 | - | 545.4 | 411.5 | 427.5 |
| R7 | 533.6 | 1966.3 | 545.4 | 2377.8 | 427.5 |
| R9 | 533.6 | 3021 | 545.4 | 3432.5 | 427.5 |
| R11 | 800.3 | - | 812.2 | | 427.5 |
| R13 | 800.3 | 3021 | 812.2 | 3432.5 | 427.5 |
| R15 | 800.3 | 1966.3 | 812.2 | 2377.8 | 427.5 |
| R17 | 800.3 | 912.2 | 812.2 | 1323.7 | 427.5 |
| R19 | 800.3 | 4075.1 | 812.2 | 448.6 | 427.5 |
| NR1 | 266.8 | - | 278.6 | | 427.5 |
| NR3 | 266.8 | 912.2 | 278.6 | 1323.7 | 427.5 |
| NR5 | 533.6 | - | 545.4 | | 427.5 |
| NR7 | 800.3 | - | 812.2 | | 427.5 |

III. RESULTS

Effect of Mg²⁺ on the Anaerobic Digestion of WAS

To reduce the concentration of NH₄⁺ in the anaerobic digester effluent through struvite formation, the concentration of Mg, NH₄ and P must be at the right ratio. Stoichiometric of molar ratios of Mg:NH₄:P ranging from 1:1:1 to 1.3:1.3:1 have been reported for varying types of sludge [12],[5]. There is no published literature on the optimum ratios for struvite formation in anaerobic digesters for domestic WAS. Considering the molar concentration of Mg²⁺, NH₄⁺ and PO₄³⁻ in the WAS used in this study (Table 1), Mg and PO₄³⁻ are the limiting species. Therefore, the effect of the addition of Mg²⁺ only or Mg and PO₄ was assessed. The addition of increased dosages of Mg²⁺ of 266.8, 533.6, and 800.3 mg/L showed little effect on the cumulative gas production (CGP), where 215.1, 218.9 and 211.2 mL of gas in total were produced in reactors R1, R5 and R11 respectively, compared to 215.1 mL CGP generated in the control reactor (Figure 1). These results indicate the presence of Mg²⁺ in the reactors at concentrations of 279 to 812 mg/L had little effect on biogas production for the domestic WAS digested in these reactors. both with and without adjusting the initial pH.

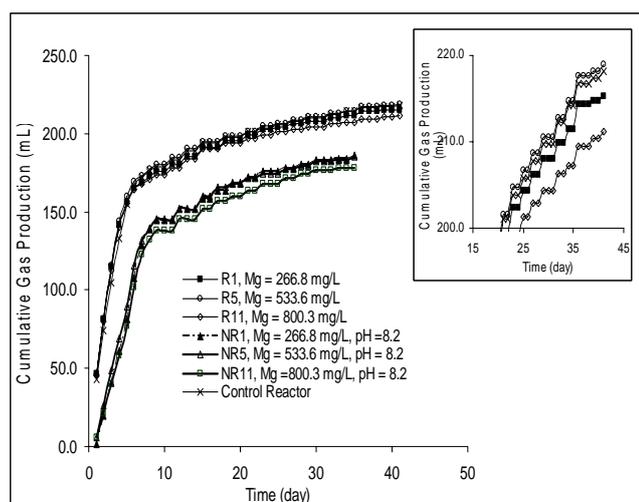


Fig. 1 CGP of biogas at increased Mg concentrations without pH adjusting and at pH = 8.2

The performance of R1, R5 and R11 in terms of effluent COD indicate slight enhanced COD removal of 10% (Table 3). This is also accompanied by an enhanced PO_4^{3-} removal of 28%, 51% and 55.7% compared to the control, but this was accompanied with an increase in NH_4^+ concentration. The trend in Mg^{2+} fixation in these reactors correlated with the enhanced PO_4^{3-} removal suggesting the formation of compounds that contain Mg and P other than struvite. The formation of newberyite $\text{MgHPO}_4 \cdot 3\text{H}_2\text{O}$ in the pH range 6.4 – 7.7 and in the presence of ammonia [13]

B. Effect of $\text{PO}_4\text{-P}$ in the Presence of Mg^{2+} on Anaerobic Digestion of WAS

As discussed above, the addition of Mg^{2+} to the anaerobic digestion reactors, lead to an increase in NH_4^+ concentration in the reactors' effluent. Therefore, to assist the formation of struvite P was supplied to the reactors. Reactors R5, R7 and R9 all received 533 mg/L Mg^{2+} , but R7 and R9 received increased P dosages of 641.6 and 958.1 mg/L, respectively. The CGP produced in reactors R5, R7 and R9 is shown in Figure 2. It is observed that the presence of $\text{PO}_4^{3-}\text{-P}$ at a concentration of 641.6 mg/L has inhibitory effect on the gas production, digestion in reactor R7 slowed down, particularly during the first 10 days, leading to 10% net reduction in CGP compared to the control. The presence of P at a concentration of 958.1 mg/L (R9) exhibited a strong inhibitory effect on gas production reflected in 75% reduction in CGP, which extended over the first 30 days of the digestion run. The reactor did not fully recover and the total CGP measured at day 41 was around 40% less than the control. The inhibitory effect observed in these reactors could be attributed both to cationic toxicity and the high concentrations of P. These results are in agreement with [14] where Mg^{2+} and Na^+ showed an IC_{50} (the inhibitory concentration of a chemical resulting in 50% reduction in microbial activity) at concentrations of 1900 mg/L. and 7000 mg/L. Although the amounts of Na supplied in R7 and R9 are 1428 and 2194

mg/L respectively, it is possible that the amounts of Na and other cations originally present in the wastewater contributed to this effect. [15] observed that the inhibitory levels of Na during anaerobic digestion were higher for an unadapted sludge compared to sludge obtained from digesters treating high saline wastewaters. The presence of cations and anions was found to have a great influence on the IC_{50} concentration of Na. For example, it was found that after 40 days of digestion, two sludge samples subjected to 6.9 and 21.5 g/L Na respectively, displayed an increase in the relative methanogenic activity from 0% to about 45% of the blank activity, which was attributed to the adaptation of methanogenic bacteria to Na and other cations and anions present in the assayed media.

The effect of the addition of P at varying concentrations and a fixed dosage of high concentration of Mg was assessed using reactors, R17, R15 R13 and R19, which all had the same background Mg^{2+} concentration of 800.3 mg/L and increased P dosages of 297.7, 641.6, 958.1 and 1329.8 mg/L, respectively (Figure 3). The CGP in these reactors showed a similar trend to that observed in Figure 2. However, an inhibitory effect was only observed in R13 and R19, that is at 958.1 and 1329.8 mg P/L compared with 641.6 mg P/L in the presence of 533.6 mg Mg^{2+} /L (R15). This suggests that reaction during the digestion lead to the formation of compounds of PO_4^{3-} and Mg^{2+} , magnesium phosphates, and the excess amounts remaining in the digester, after the completion of these reactions, is likely responsible for the observed inhibitory effect.

Reactor R3 received dosages of Mg and P of 266.8 and 297.7 mg /L respectively, to bring about soluble $\text{Mg:NH}_4\text{:PO}_4$ in the reactor to a molar ratio of 1:1:1. Similarly, reactors R5 and R11 received MgCl_2 and Na_3PO_4 to achieve molar ratio of $\text{Mg:NH}_4\text{:PO}_4$ of 2:1:2 and 3:1:3 to assess performance at an initial equi-molar ratios of Mg and P. The CGP in reactor R3 was around 3% less than the CGP collected from the control reactor (Figure 4). It is also observed that excess amounts of P have an inhibitory effect on the digestion even when added at a stoichiometric equi-molar ratio to Mg. For example, the presence of increased moles of and P slowed the rate of gas production during the first 15 days, an effect that was more pronounced for the high molar ratio of Mg:N:P of 3:1:3 (R13) compared to 2:1:2 molar ratios (R7) (Figure 4). The total CGP in reactors R7 and R13 decreased at 10% and 20%, respectively, compared to the control reactor Considering that Mg dosages of 266.8 to 800.3 mg/L had slight adverse effect on CGP (Figure 1), it is likely this inhibition is due to the presence of P at concentrations equal to or higher than 641.6 mg/L for the WAS used in these reactors. In addition, the results in Figure 3 and Figure 4, suggest that the inhibitory effect is more influenced by the presence of high P levels than that of Na concentration.

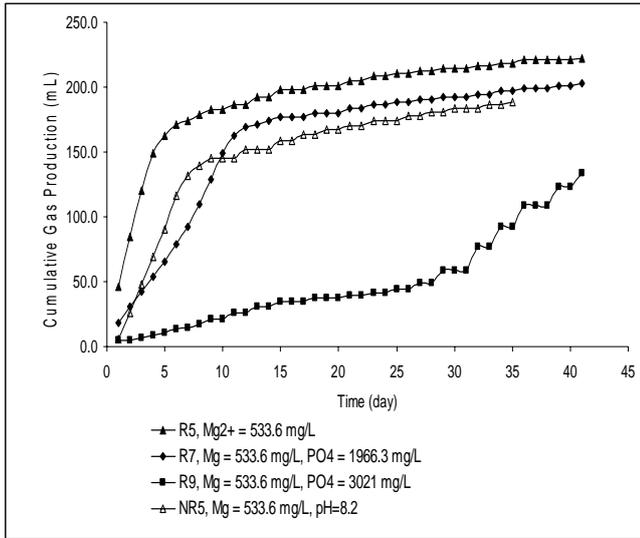


Fig. 2 CGP for increased levels of PO_4^{3-} and Mg^{2+} concentration of 533.6 mg/L

C. Effect of pH on Anaerobic Digestion of WAS

The pH in R1, R5 and R11 measured at day 41 was 7.06, 7.03 and 6.97, respectively. Although anaerobic digestion reactors perform best within a pH range of 6.8 – 8.0 struvite formation has been noted to occur over a wide pH range (7-11) but the optimum struvite formation has been reported at pH of 8.7 – 9.3 (Nathan et al 2003, Wang et al 2005). To assess the effect of increased pH on the WAS tested in this study, duplicate reactors of R1, R5 and R11 namely NR1, NR5 and NR11 were operated at an initial pH of 8.2. Figure 1 shows that adjusting the initial pH = 8.2 lead to 15.3%, 15.2% and 17.8% reduction in CGP in NR1, NR5, NR11 respectively.

The CGP in reactors NR3, with $Mg:NH_4:PO_4$ at a molar ratio of 1:1:1 indicate a strong inhibition compared to that observed for R1, R5 and R11. This inhibition is reflected in slow gas production compared to the reactors that did not receive additional supplies of P from an external source, where 150 mL of GP was achieved at day 5 in R3 compared to 20 days for NR3. These results indicate the presence of Mg^{2+} in the reactors at concentrations of 279 to 812 mg/L had little effect on biogas production for the domestic WAS digested in these reactors both with and without adjusting the initial pH.

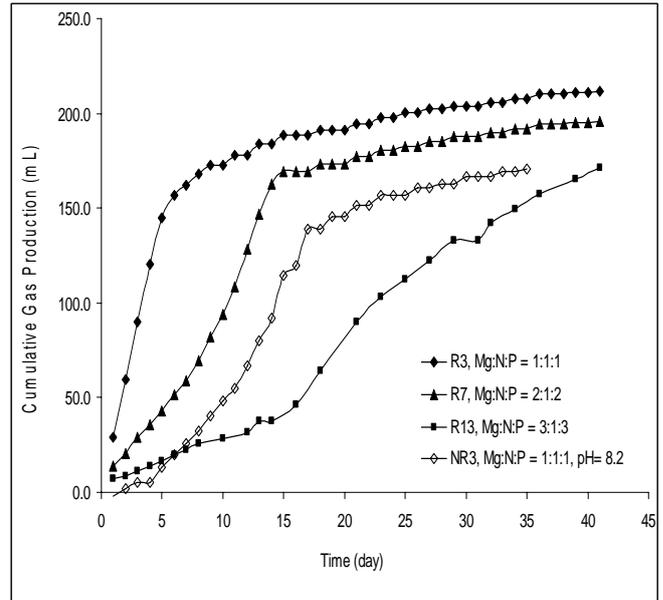


Fig. 3 CGP for varied ratios of $Mg:NH_4 : PO_4^{3-}$

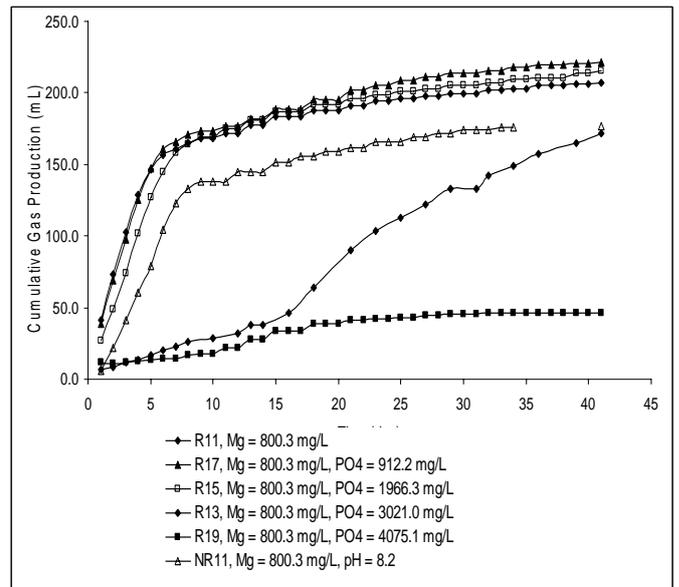


Fig. 4 CGP for increased levels of PO_4^{3-} and Mg^{2+} concentration of 800.3 mg/L

D. Anaerobic Digestion Reactors Performance

The digested WAS COD for the reactors that received a dosage of Mg, but no external supply of P (R1, R3, R5, and NR1, NR5 and NR11) showed enhanced COD removal. However this was associated with 22.4% – 26.2% increase of NH_4^+ in the reactors' effluent (Table 3). The results suggests the formation of compound(s) constituting Mg and PO_4 (i.e. magnesium sulphates), consequently ammonia generated remains in the reactor.

It is also observed that the presence of excess P in the AD reactors has a strong inhibitory effect on WAS digestion in terms of COD removal and CGP performance. For example for R9 and R19 the addition of Mg:P at 533.6:958.1 and 800.3:1392.8 in reactors R9 and R19 lead to a drop in CGP to 129 and 46 respectively compared to 218 mL for the control.

The results shown in Table 3 indicate reactor R15 had the best performance in terms of CGP, COD removal and NH_4^+ levels in the reactor's effluent. The reactor had $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$ at a molar ratio of 1.28:1:00:1:0. Reactor 17, which received $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$ at 1.28:1:00:0.60 also showed a slight enhancement in performance, where 2.1% increase in COD removal and 5.4% less NH_4^+ in the effluent were achieved. The results are in agreement with those reported by [16] and [5] who found that a molar ratio of Mg:P of 1.3:1 was required to guarantee phosphorus removal as struvite. Authors of [5] also reported that at a molar ratio of 1:05:1 Mg:P a mixture of struvite and hydroxyapatite was obtained. These variations were found to be related to the concentration of calcium hence competing with Mg for P.

Therefore, further research into the potential of N and P removal during the anaerobic digestion process where the composition of the influent sludge for Ca and other constituents is assessed as part of a process to optimise N and P removal.

The XRD analysis for the AD reactors confirmed the formation of struvite, as shown in Figure 5A and 5B for selected reactors, R9 and NR7 respectively. The concentration of halite (assuming that the counts (y-axis) are proportional to the constituent concentration) seems to correlate with the dosage of $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$ in the reactors, for example R9 and R13 (not shown) received the same dosage and showed the same counts. Similarly, a proportional relationship was observed for R19 and R4 (not shown). It is also observed that reactors which did not receive any $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$ loading had halite as a constituent in the sludge (Figure 5B). This indicates the presence of Na in the WAS and seed which may have contributed to the inhibition to COD removal and CGP in these reactors.

TABLE III
CHANGES IN Mg^{2+} , $(\text{NH}_4^+)_s$, $(\text{PO}_4^{3-})_s$ AND AFTER 41 DAYS

| Reactor | | Effluent Mg^{2+} (mg/L) | Mg Fixing (mg/L) | Mg Fixing (%) | COD (%) | $(\text{NH}_4^+)_s$ Increase (%) | $(\text{PO}_4^{3-})_s$ Removed (%) | CGP (mL) |
|---------|-----------|----------------------------------|------------------|---------------|---------|----------------------------------|------------------------------------|----------|
| Control | | 9.8 | 2.1 | 17.5 | | 65.4 | 34.63 | 215.1 |
| R1 | Mg, No P | 97.0 | 181.6 | 65.2 | 9.9 | 91.6 | 62.58 | 208.2 |
| R3 | 1:1:1 | 11.0 | 267.6 | 96.1 | -7.1 | 74.7 | 63.28 | 210.6 |
| R5 | dMg, No P | 209.6 | 335.8 | 61.6 | 3.5 | 91.0 | 85.66 | 218.9 |
| R7 | 2:1:2 | 6.9 | 538.5 | 98.7 | -32.6 | 58.9 | 67.45 | 202.5 |
| R9 | d Mg, T P | 4.9 | 540.4 | 99.1 | -51.6 | 70.9 | 15.22 | 129.0 |
| R11 | TMg, No P | 339.3 | 472.9 | 58.2 | 9.9 | 87.8 | 90.28 | 211.2 |
| R13 | 3:1:3 | 6.5 | 805.7 | 99.2 | -61.6 | 25.7 | 66.79 | 189.3 |
| R15 | T Mg, d P | 24.8 | 787.4 | 97.0 | 8.1 | 42.0 | 84.90 | 215.1 |
| R17 | TMg, P | 179.9 | 632.2 | 77.8 | 2.1 | 60.4 | 94.49 | 219.5 |
| R19 | T Mg, 4P | 10.9 | 801.2 | 98.7 | -92.0 | 92.0 | 10.0 | 46.10 |
| NR1 | Mg, No P | 116.7 | 161.9 | 58.1 | 12.1 | 89.9 | 0.89 | 184.7 |
| NR3 | 1:1:1 | 2.3 | 276.3 | 99.2 | -9.2 | 66.5 | 0.66 | 166.4 |
| NR5 | dMg, No P | 288.1 | 257.2 | 47.2 | 22.7 | 83.4 | 0.88 | 185.0 |
| NR11 | TMg, No P | 450.4 | 361.8 | 44.5 | 14.2 | 88.8 | 0.86 | 177.1 |

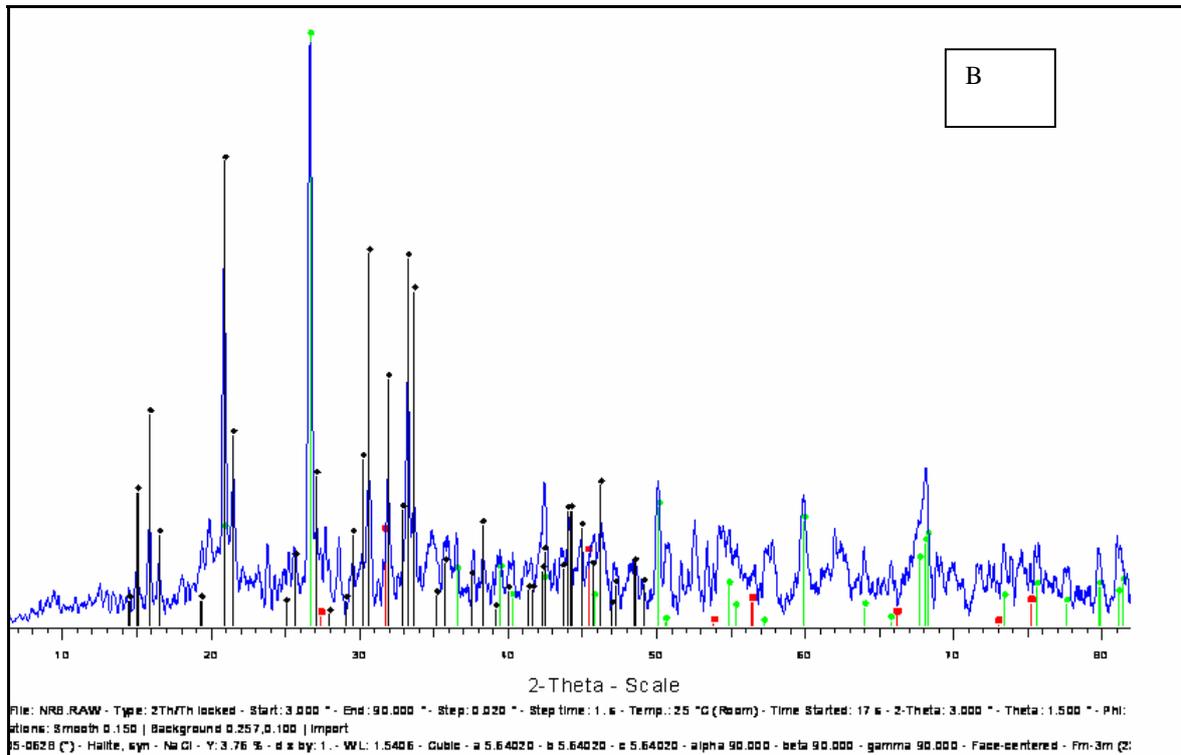
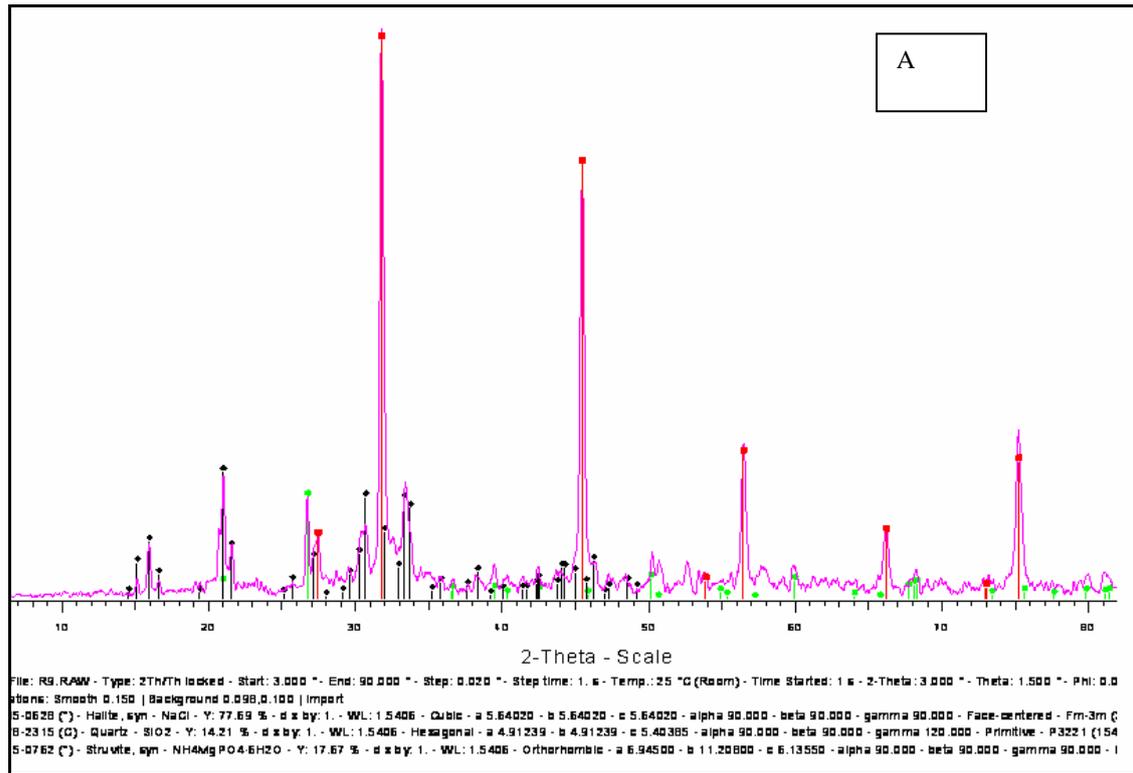


Fig. 5 XRD analysis of digested WAS A) R9 and B) NR7

V. CONCLUSION

- Batch anaerobic digestion tests showed that Mg²⁺

concentration in the range 279 – 812 mg/L had insignificant effect on CGP but lead to a slight increase in COD removal.

- The presence of excess PO_4^{3-} indicated an inhibitory effect on CGP and COD removal. The inhibitory concentration decreased with increased background Mg^{2+} concentration, where P dosages of 958.1 and 1392.8 in the presence of Mg^{2+} at 533.6 and 800.3 mg/L respectively, had a strong inhibitory effect on the digestion process.
- The reactor that had soluble $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$ at a molar ratio of 1.3:1:00:1.0.1 achieved the best performance enhancement of 8% increase in COD removal and 32% reduction in NH_4^+ in the reactor supernatant.
- Overall, the results show that there is a potential to optimise conventional anaerobic digestion such that supernatant lean in P and N, and sludge rich in nutrients are obtained.

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