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ORIGINAL RESEARCH

Recovery of Agro-based Bio-Products from Cattle Farm Waste Using Aerobic Fluidized Crystallization Reactor

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ABSTRACT

Plenty of well proven anaerobic methods are in existence to leisurely convert cattle farm waste into useful derivatives such as manure. But, unfortunately, anoxic processes are hectic and quite hard to comply with. Thus, the present study aimed to primarily address high-end solutions to overcome the technical drawbacks of the existing anaerobic methods. Nutrients from the cattle farm waste streams were recovered by aerobic treatment mechanism, utilizing fluidized bed reactor (FBR) with dairy and goat manures as feed influents. In order to optimize the recovery, an effective pH value was determined and upheld by using a buffer solution of $MgCl_2 \cdot 6H_2O$ and NaOH. The maximum amount of struvites recovered from goat wastes was 93.2% of magnesium, 70.2% of ammonia, and 92.2% of phosphate. While the witnessed recovered fractions were scanty in the case of dairy wastes, which were as follows: 92.2% of magnesium, 68.2% of ammonia, and 91.2% of phosphate. The influence of pH and HRT on the yield of nutrient recovery has played a significant role. The study revealed supplementary recovery was achieved at the optimum condition (i.e., pH 9.0, HRT 2h) when the influent stream was doused with 0.06 M Mg_2^+ . Ultimately, it was concluded that goat manure has a higher capacity of removing the nutrients compared to the dairy manure.

KEY WORDS: *Eutrophication, fluidized bed reactor, nutrients, recovery, struvite*

INTRODUCTION

With the increase in the global population and the net income per person, there is a need to increase the agricultural yield, which can be accomplished by the increased use of fertilizers. In the present scenario, half of the world population relies on synthetic fertilizers to obtain their food resource (Annicchiarico *et al.* 2011). However, these conventional chemical fertilizers can be replaced by eco-friendly cattle farm manure if it is properly recovered from the mixed waste consortium. The conventional approaches for discarding livestock manure comprises direct

land application, disposal of the nutrient-rich wastewater on cropland, and dispersion by means of dilution in the receiving water bodies (Beal *et al.* 1999; Yetilmezsoy *et al.* 2009). The above practices may lead to the loss of nutrients and vigorous environmental interventions. The most common issues associated with the disposal of untreated manure are the release of obnoxious gases, odor generation, and discharge of nutrients and pathogens that may cause eutrophication and affect human health (Burns *et al.* 2001; Bouropoulos and Koutsoukos 2000).

In 1997, 65% excess nitrogen and 68% phosphorous were estimated to be generated in the US (Ribaudo *et al.* 2003). Ammonia in the form of nitrogen may lead to numerous air and water quality problems.

A wide range of wastewater streams, including waste generated from swine farms (Burns *et al.* 2001; Nelson *et al.* 2003) and waste streams from agricultural industry (Altinbas *et al.* 2002), have been taken into consideration for nutrient recovery using struvite crystallization. Nitrogen, phosphorus, and magnesium can be recovered from the waste streams by precipitation of minerals as struvite ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$). Struvite recovery meets the legal requirements imposed on wastewater disposal and reduces the area needed for wastewater application. The yield of struvite crystallization relies on several parameters such as the pH and temperature of the solution, chemical composition, degree of saturation in terms of major nutrient fraction, existence of erstwhile ions like calcium, ionic strength of the waste stream, etc. Currently, when the global population is working on developing sustainable ecofriendly substitutions of chemical fertilizers, struvite crystallization has proven to be one of the most reliable practices to recover nutrients efficiently from cattle waste. Battistoni *et al.* (1997) and Bhuiyan *et al.* (2008) recovered phosphorous using fluidized bed reactor in the absence of alkaline chemicals. CO_2 stripping was proven to be sufficient to reach the operating pH and there was no requirement of additional calcium or magnesium salt as the process was anoxic the pH condition of both the experiment found to be marginally contradicting with the present research. Though the use of Ca or Mg amplified supernatant formation but did not prevalent the efficiency of the process. Whereas, the study undertaken by Guadie *et al.* (2014) has reported almost equivalent observations in terms of optimal operating conditions with and without the inclusion of cone, though the recovery reported from the waste stream was approximately near 80% in both the cases which is slightly lower when compared to the present work. A study undertaken by Jaffer *et al.* (2002) recorded higher removal of phosphates up to 97% from the waste stream when a balanced ratio of 1.05:1 was maintained in the influent condition, otherwise a sharp declination in the yield was observed and it's quite significant to state that maintaining such a condition in field scale is a hectic job. Moreover, a review work carried out by Katakai *et*

al. (2016) has explicitly mentioned the techno-economic issues attributed to the large scale economic extraction of phosphate manure from numerous waste streams via anaerobic segregation and highlighted the necessity of addressing the research gaps. The feasibility of pilot scale struvite crystallization was assessed in a study conducted by Schuiling and Andrade (1999) in the Netherlands with the help of continuously stirred tank reactor and the yield of the same found to be satisfactory irrespective of any logical output explanations and the same researchers also reported a detrimental impact on recovery in the case of higher suspended solid concentrations which is not a matter of concern for FBR. In the case of a double saturation model, efficiency foremost relies on sand contact time and pH. Phosphate can be obtained using fluidized bed reactor agitated with air (Ueno and Fujii 2001).

Therefore, the preliminary aim of the present study was to investigate and propose an up-scalable extraction mechanism for struvite which is readily adoptable irrespective of the influent stream and inexpensive.

RESULTS AND DISCUSSION

To evaluate the reactor performance, recovery efficiencies were calculated on the basis of phosphorus removal from cattle farm wastes (Pastor *et al.* 2008). Characteristics of diluted dairy manure and goat manure are presented in Table 1. In order to consider the recovery efficiency for goat and dairy manure wastes, the precipitation efficiency was calculated based on the total phosphorus concentrations in the influent and effluent streams (Le Corre *et al.* 2009; Salminen *et al.* 2001; Tan and Lagerkvist 2011; Oscar *et al.* 2013). When fluidized bed reactor was operated under optimal conditions of pH (9), HRT (2 h), Mg:P (1.25), and N:P (7.5) for goat and dairy farm wastes, the phosphorus precipitation efficiency was determined as 92% and 91%, respectively.

In the present study, some of these factors were observed for assessing the feasibility of FBR for goat and dairy farm wastes. Under various experimental conditions, the reactor showed better phosphorus removal efficiencies between 60% and 92% compared to the nitrogen removal efficiencies between 30% and 70%.

Table 1 Characteristics of Diluted Dairy and Goat Manure

<i>Parameters</i>	<i>Concentrations for dairy manure*</i>	<i>Concentrations for goat manure*</i>	<i>Unit</i>
pH	7.48	8.08	-
Ammonia	1.47	1.68	mg/L
Phosphate	4	6	mg/L
Total Dissolved Solids	33.3	28.3	mg/L
Total Solids	56	66	mg/L
BOD	28	36	mg/L
COD	196	246	mg/L
Total Hardness	462	498	mg/L
Calcium Hardness	325	365	mg/L
Chlorides	70	65	mg/L
Magnesium	180	168	mg/L

**subjected to field conditions of the influent waste stream*

The most co-relatable reason behind the significantly lesser recovery of nitrogen is of its presence in various forms such as NH₃-N, Kjeldahl Nitrogen, and total Nitrogen and all the forms are not typically possible to subject for equal recovery under the same reactor environment. The above-recovered fraction showed a similar range of recovery, as stated by Oscar *et al.* (2015). This has also been reported by many other previous studies (Uludag-Demirer *et al.* 2005; Zhang *et al.* 2010; Huchzermeier and Tao 2012; Uysal and Kuru 2013). The effect of pH in the removal of phosphate and magnesium was determined for both the animal wastes. When the reactor was working at optimal pH > 9.0, more than 90% removal of phosphorus and magnesium was achieved for both wastes. The removal of ammonia was less (i.e. around 35%) compared to that of phosphorous and magnesium in both types of wastes. According to the previous literature, the most suitable operating range of pH for struvite crystallization varies between 8 and 10 (Fattah *et*

al. 2008; Huchzermeier and Tao, 2012) Fig. 1(a) and 1(b) show the removal efficiencies of both farm wastes under different pH values.

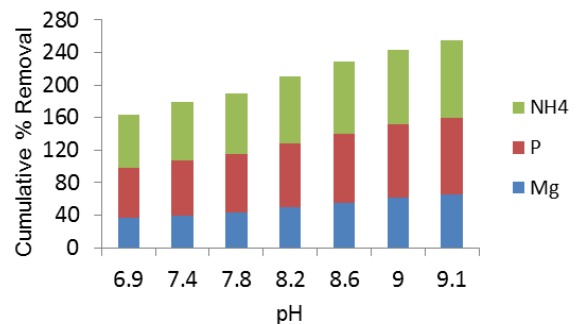


Figure 1(a): Effect of pH on Nutrient Removal Efficiency for Goat Manure

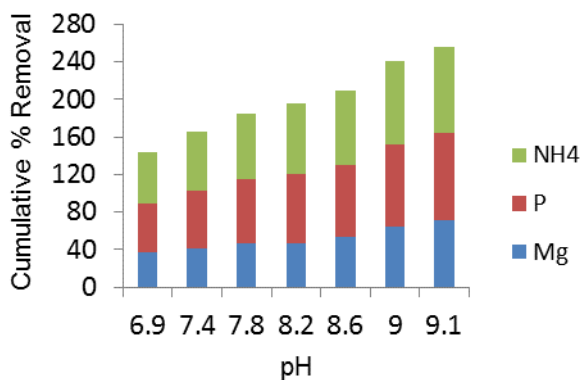


Figure 1(b): Effect of pH on Nutrient Removal Efficiency for Dairy Manure

The reactor was operated at continuous HRTs of 1, 2, 4, 8, 10, and 12 h, and the result showed more than 70% of phosphorus and 35% of nitrogen removal efficiencies in the first 1 h HRT as shown in Fig. 2. The effect of HRT on removal efficiencies was found to be insignificant. For instance, when the HRT increased from 2 to 12 h, 90 and 92.5% removal efficiencies were observed simultaneously due to higher micro-organic activities. Fig. 2(a) and 2(b) showed the experimental results resemblance with the values obtained by preceding studies (Battistoni *et al.* 1997; Le Corre *et al.* 2007). According to Le Corre *et al.* (2007), the removal efficiencies obtained after 1 and 24 h were similar (79%). An enhanced phosphorus removal before HRT of 1h was observed, indicating the insignificance of HRT in the removal efficiencies of phosphorous (Liu *et al.* 2008).

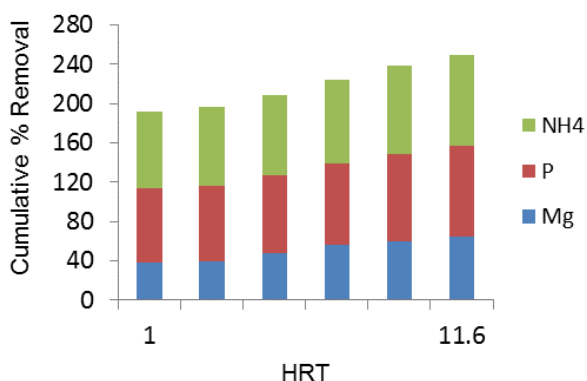


Figure 2(a): Effect of HRT on Nutrient Removal Efficiency for Goat Manure

Correlation between the concentration of Mg₂⁺ and equilibrium pH

Equilibrium conditions have been accomplished for all the experiments based on the fixed pH values. Struvite formation kinetics occurs within 1 h, as mentioned by Suvathika and Ashok Kumar (2016). The variation in the pH value of the solutions also shows a similar pattern throughout the experimental period.

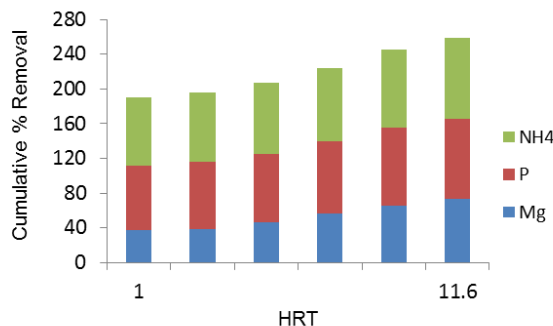


Figure 2(b): Effect of HRT on Nutrient Removal Efficiency for Dairy Manure

In order to adjust the initial solution pH for both the manures, NaOH was utilized based on its reaction time, shown in Fig. 3(a) and 3(b). Eun *et al.* (2015) stated that the most effective pH value required to precipitate the maximum amount of nutrient is ~12.0. The above mentioned pH value showed little higher margin compared to the values found to be effectual for the present study. This may be due to the different influent sources used for this study.

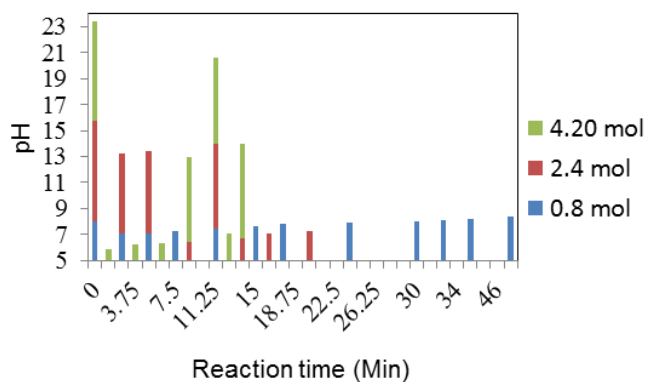


Figure 3(a): Adjustment in the influent waste stream pH of Goat Manure during the Experimental period

In agreement with our study, other authors like Lee *et al.* (2010), Rahman *et al.* (2011), Ye *et al.* (2011), and Hutnik *et al.* (2013) have also concluded that an alkaline environment

with the pH value of 8 to 12 is the most suitable for struvite crystallization.

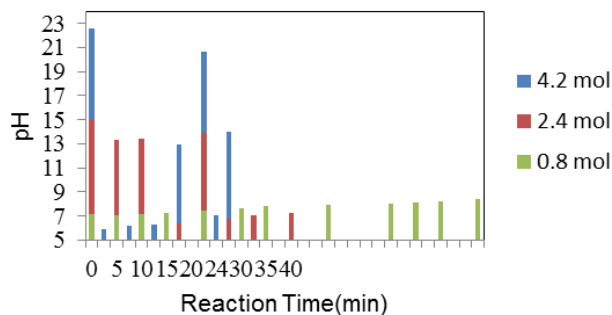


Figure 3(b): Adjustment in the influent waste stream pH of Dairy Manure during the Experimental period

When the reactor was doped with $MgCl_2 \cdot 6H_2O$ and $NaOH$, there was an uninterrupted and steady increment in pH as long as the equilibrium conditions were sustained. However, when only $MgCl_2 \cdot 6H_2O$ was supplied as input, the pH remained constant at ~ 7.0 due to the acidic impacts of $MgCl_2 \cdot 6H_2O$ (Fig. 3c and 3d).

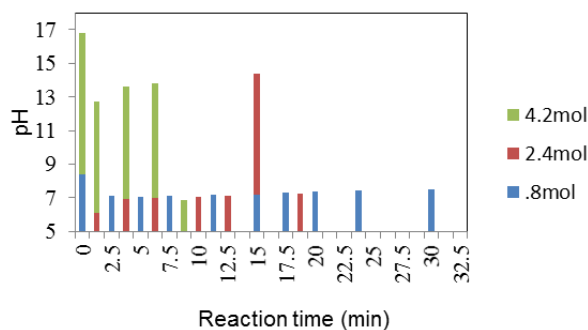


Figure 3(c): pH variation for Goat Manure during the Experimental period

The equilibrium pH in the reactor doped with $MgCl_2 \cdot 6H_2O$ for dairy as well as goat manures, as shown in Fig. 5(a) and 5(b), respectively. The pH and the concentration of Mg_2^+ ions showed an inversely proportional relation when $MgCl_2 \cdot 6H_2O$ was used; this was because of the increased acidity, instigated by the Cl^- ions in the solution. Hence, the removal of NH_4^+ as ammonia was not considered for the reactor for both the manures. However, Doyle and Parsons (2002) and Yetilmezsoy and Zengin (2009) stated that $MgCl_2 \cdot 6H_2O$ and $MgSO_4 \cdot 6H_2O$ can be used as the buffer solution to maintain the pH.

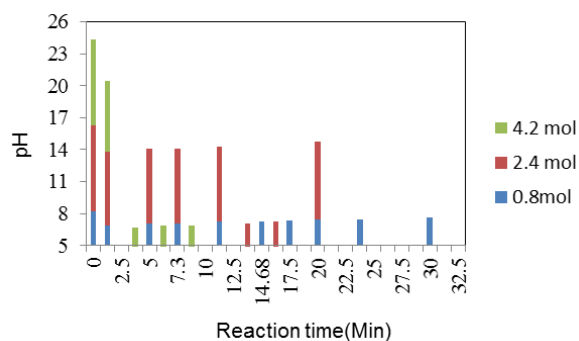


Figure 3(d): pH variation for Dairy Manure during the Experimental period

But the use of $MgCl_2 \cdot 6H_2O$ alone may form $Ca_3(PO_4)_2$, which further suppresses the formation of struvite crystals.

Correlation between the concentration of Mg_2^+ and the removal efficiency of NH_4^+

A molar ratio of magnesium ammonium phosphate greater than 1:1:1 was the most prominent for struvite hard crystal formation (Yilmaz and Demirel 2008). In this study, various molar ratios were tested to ensure optimum yield, and all the trails were run by maintaining a higher concentration of PO_4^{3-} . The molar ratios of $Mg_2^+ : NH_4^+ : PO_4^{3-}$ were in the range of 0.6:1.0:4.2 to 3.6:2.1:0:4.8, and 1.1:1.0:10.0 was carried out. The remaining concentrations of NH_4^+ at equilibrium when the reactor was doused with different concentration of Mg_2^+ for different manures are shown in Fig. 4 (i.e. with and without pH adjustment).

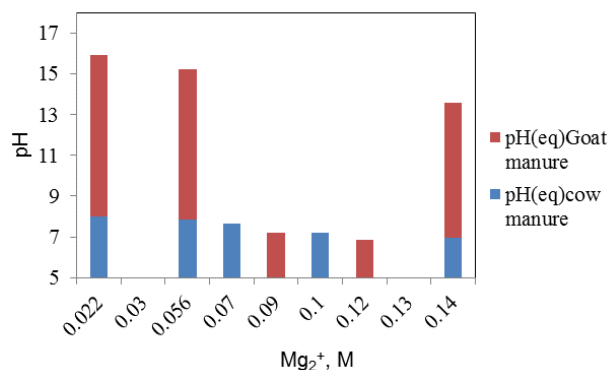


Figure 4: The Equilibrium pH in the Reactors Dosed with Different Mg_2^+ Ions

The NH_4^+ concentrations varied inversely with Mg_2^+ concentration despite the initial adjustment of pH and the Mg_2^+ ion source.

The equilibrium concentration lesser than 10 mg/L was maintained for NH_4^+ by doping $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ at the rate of 3.0 mole per mole of NH_4^+ for dairy as well as goat manures. This concentration was larger than the quantity required for stoichiometric struvite formation.

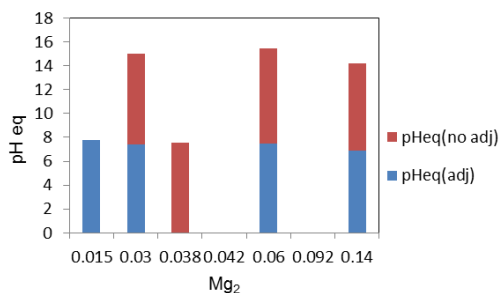


Figure 5(a): The change in Equilibrium pH of the reactor dosed with $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ for Goat Manure

The concentration of Mg_2^+ was greater than the stoichiometric amount required to accomplish the preferred concentration of NH_4^+ in the effluent stream, which varies according to the composition of the influent wastewater.

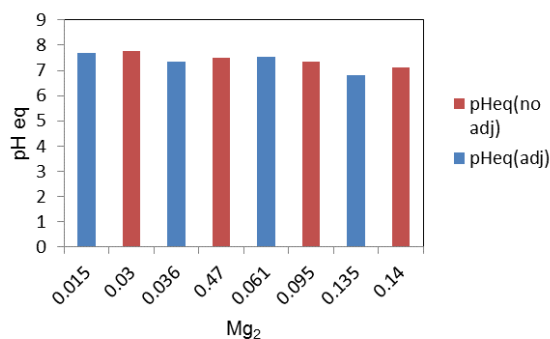


Figure 5(b): The Equilibrium pH in the Reactor Dosed with $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ for Dairy Manure

The amount of Mg_2^+ required for the experiment and the conditional solubility of struvite depends majorly on the existence of complexing agents (which formed complexes with Mg_2^+), the equilibrium pH, and the ionic strength of the influent wastewater. Preliminary pH alteration to 8.50 by using NaOH as a buffer solution was performed to nullify the impact of initial pH fluctuations on the removal of NH_4^+ . The enduring concentrations of NH_4^+ in the bioreactors with an initial pH of 8.50 did not fluctuate drastically from that in the reactors without pH adjustment (pH 7.45–7.93) as in Fig. 5(a) and 5(b). Oscar *et al.* (2015) reported different fractions

of ammonia recovery, such as 30%, 53% and 55% for successive pH values of 11, 12 and 13, which showed minimal interference with the values when compared to the recovered percentage obtained for the present study.

This indicates the meager variation took place in the preliminary pH of the effluents collected from the reactor and did not influence the efficiency of the NH_4^+ removal by struvite crystallization for both the manures.

The removal efficiency for NH_4^+ from the waste stream was achieved to be above 85% while the concentration of Mg_2^+ was greater than 0.06 M for both cattle farm wastes despite the addition of the source of Mg_2^+ , as shown in Fig. 6.

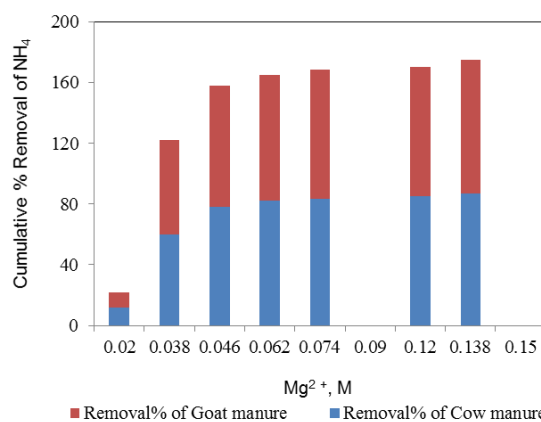


Figure 6: NH_4^+ Removal Efficiency from the waste stream

CONCLUSION

After enormous process enhancement and fulfilling of the pertaining technical laggings, the maximum phosphorus removal efficiency was observed to be more than 90% under the optimal operating conditions, independent of HRT values. The average ammonia and phosphorus removal efficiencies were found to be 40% and 80%, respectively. This indicates that HRT did not influence nutrient removal, though pH has a significant impact on the recovery of MAP. The ultimate finding indicates struvite crystallization, which slowly releases precious fertilizers in an eco-friendly way, in addition to 0.06 M Mg_2^+ for the influents of the reactor for both manures.

MATERIAL AND METHODS

Reactor Detailing

The research work involves aerobic fluidized bed struvite crystallization reactor for the extraction of MAP which was fabricated in the workshop premises, using internal recourses and acrylic material. The lab scale reactor portrayed an optimum working volume of 4 L, which comprises two prime functional segments: (1) a down-flow cylindrical tube made up of translucent PVC duct of 150 cm length, 50 cm internal diameter, and 4 L working volume that acts as a fluidization unit, and (2) a conical duct made up of identical material with 45 cm length and 20 cm internal diameter and having a bottom outlet that acts as a zone of active settlement (Lahav *et al.* 2013). In order to prevent the precipitates to get accumulated in the settling zone, two discrete units were linked by means of a PVC funnel (6 cm outer diameter and 15 cm height).

The designed prototype reactor was cylindrical in shape and predominantly conical at the bottom end. The reactor comprised two distinctive parts; the upper segment resembled a hollow cylindrical tube and the bottom segment was an inverted cone-shaped structure. Due to its conical shape, the lower part of the reactor functioned as a settling region. An outlet was provided at the lowermost portion of the FBR to remove sludge, wastewater, and struvite crystals (Fig. 7). In order to ensure the optimum mixing of the influent cattle farm wastewater and chemicals (i.e. $MgCl_2$ solution and NaOH), a peristaltic pump (Miclins, India) was provided. The reactor was operated at different pH ranges between 7.2 and 9.6 and different HRTs ranging from 1 to 12 h. It was observed that inadequate dosage of magnesium diminished the MAP removal efficiency. The pH of the solution was incessantly monitored until the equilibrium conditions were achieved; this was established by a constant value of pH with an optimum fluctuation window of 0.01 pH units.



Figure 7: Overview of the prototype lab scale reactor

A mechanical aerator was configured at the middle of the bio-reactor for better aeration of influents and to maintain an aerobic environment. An overflow outlet was designed at the upper segment of the reactor to encounter overflow wastewater when the reactor operates in a continuous flow mode. The mixing and uniformity of the wastewater concentration were achieved by means of initial vibration.

In the present study, a comparatively higher initial total suspended solid concentration (TSS) was favored during the reactor start-up period (i.e., TSS > 20 g/L) to facilitate assorted struvite crystallization during the experimental period. To accelerate the optimization of the struvite formation, the obligatory Mg:P ratio of the influents to the bioreactor and the invariable pH were retained throughout the study period. This experiment ascertained the influence of pH values and HRT in the recovery of considerable amount of nutrients.

Sampling and analyses

For chemical analysis, influent and effluent samples were collected every two days from the feed tank and external recycler outflow, respectively. The constituents of phosphorus (P), ammonium (NH_4), and magnesium (Mg) were analyzed following the standard methods (APHA, 1998). P, Mg and NH_4 were analyzed using a UV-visible spectrophotometer (UV-vis 2100, Hitachi, Japan), whereas Mg and Ca were analyzed using a flame atomic absorption spectrophotometer (Systronics, India).

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