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Corn starch industry wastewater pollution and treatment processes- A review

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Abstract

Corn starch industry contributes almost 12% of starch production. Maize starch, produced worldwide, contributes huge amount of acidic effluent (pH 3-5) containing high Chemical oxygen demand (COD) (10000-30000 mg/L), biological oxygen demand (BOD) (4000-8000 mg/L), nitrogenous pollutant (400-900 mg/L) and other pollutants. Conventional methods of anaerobic digestion and nitrification-denitrification process are widely being used to treat starch industry effluent. The anaerobic digestion requires neutral pH operation thus increases operational cost. Similarly, nitrification and denitrification processes are lengthy processes consuming high operational cost and require secondary treatment for generated excess sludge. Several technologies like low pH methanogenesis, anaerobic ammonium oxidation, and sludge pyrolysis are the newer concept found to be very promising. But it still require evaluation for effective removal of waste from corn starch industry effluent; as well as a matter of extensive research itself because of the non-confirmative bacterial characteristic, occurrence, growth factor, culture and isolation possibilities, which are still to be explored. This paper reviews the newer possibilities to treat effluent under low pH and possibilities for effective anaerobic removal of nitrogenous pollutants and incorporation on zero discharge close loop technology in corn starch wastewater treatment.

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Introduction

Starch is widely used in food, pharmaceutical, paper & textile industry in large quantities. Maize is used as a bulk source of starch production in various countries. It is the highest produced cereal crop and widely cultivated throughout the world, counts among major contributors of raw material in bulk industrial scale (Cerquiglini *et al.*, 2016). Corn starch production due to its high profitability is growing at a cumulative agronomic growth rate (CAGR) of 5.5% annually (Ficci, 2014). The first growing global market of maize in financial year 2015-16 is represented in Table 1. Starch from maize is extracted through dry milling and wet milling process in bulk scale. Corn starch and its derivatives are produced by wet milling and processing involves a series of process from screen grain, cleaning, grinding, separation of protein and starch, drying and further processing to yield derivatives, like liquid glucose, dextrose mono hydrate etc. with acid or enzymatic hydrolysis. A schematic of whole process is represented in Fig. 1.

Table 1. Global maize production 2014-2016 (Cerquiglini *et al.*, 2016)

	Maize production (mMT)		
	2014	2015	2016
USA	377.6	367.2	382.6
China	225.2	234.5	229.7
EU	171.7	150	160.7
Brazil	82.9	88.3	83.9
Argentina	39.9	42.4	45.4
Russian Fed.	42.4	39.5	41.8
India	43.1	38.3	41.7
Ukraine	39.77	33.4	36
Mexico	31.8	32.8	31.4
Canada	22.1	25.7	25.8
Nigeria	19.5	19.2	20.2
Indonesia	19	19.4	19
Ethiopia	19.2	16.6	17
Turkey	12.9	15.1	14.2
Australia	11.3	12.6	12.5
Other	179.5	168.6	162.6
World	1337.7	1303.6	1324.5

Wet milling process is absolutely water dependent process and require huge amount of water. In any standard corn starch industry each metric ton of starch grinding requires almost 5-11m³ of water and so produces huge amount of wastewater(Jackson and Shandera, 1995). The generated effluent is acidic(pH 3.0-5.5) by nature with high chemical oxygen demand (COD 6000-19000 mg/L) (Ran *et al.*, 2014).

The larger part of this effluent contains biologically degradable components and possesses high biological oxygen demand (BOD) around 4000-9000 mg/L (Dubey, 2006). The wastewater after corn processing contains high amount of volatiles, precipitation of modified starch, dissolved chemicals used in modification, impurities from corn syrup, gluten and dextrose and characterized as high strength (Cancino-Madariaga and Aguirre, 2011).

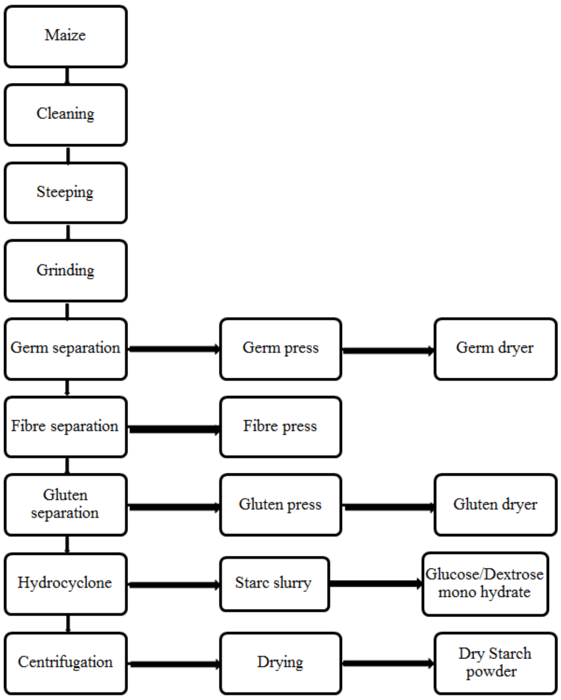

Fig. 1. Schematic of corn starch production by wet milling method.

Due to high biodegradability biological treatment of this effluent is widely practiced by several available technologies to protect the environment and to comply with pollution control guidelines (Eu, 2010). The main goal of all available technologies are to remove maximal removal of carbonaceous and nitrogenous contaminant from water and to make the water reusable for irrigation purpose or to make it potable by applying further advanced technologies like reverse osmosis after biological treatment (Cancino-Madariaga and Aguirre, 2011).

The technologies developed over time for aerobic and anaerobic treatment are in consideration to neutral pH waste treatment, while starch effluents are highly acidic in nature.

Thus there is a compulsory requirement to neutralize the effluent by dilution or chemical addition before introducing it to treatment system (Sklyar *et al.*, 2003).

Anaerobic treatment earlier was reported to happen between pH 6.8-7.6 (Chan *et al.*, 2009). But recent development to understand methanogenic reaction reported multiple cases where biological degradation of nutrient was observed very prominently in low pH ranging 3.3 to 5.5 (Savant *et al.*, 2002). Also low cost treatment like ANAMMOX technology proved to be highly efficient removing nitrogen load from wastewater. The introduction of these phenomena to large scale treatment system will tremendously change the cost effectiveness and acceptability of biological treatment amongst food processing industries.

The purpose of this review work is to understand present status of starch industry pollution contribution to water bodies and to review pros and cons of the various technologies associated to treat starch industry effluent. Also this work will focus on definite scope of research to address the present technological drawback, in order to ensure reduction of treatment cost and efficiency.

Severity analysis of effluent and biochemical evaluation for suitable treatment option

Corn starch processing in most recent trend follows close loop technology to reduce wastewater generation by inter-circulation of process water at various steps like multistage steeping, gluten separation, fibre separation etc. (Cancino-Madariaga and Aguirre, 2011). Volumetric estimation of wastewater generation in each step of corn starch production for a standard 150 MT maize crushing plant is shown in Table 2. The wastewater generated in every step contains high amount of suspended solid as biodegradable material, COD, sulphate, phosphate and chloride load. A trend analysis of process water chemical characteristic is represented in Table 3

Table 2. Water balancing and wastewater generation in corn star production.

Process	Water requirement (m^3)	Recirculation (M^3)	Wastewater generation (M^3)
Corn steeping	750	150	20
Gluten decantation	600	300	300
Starch decantation	500	200	100
Ion exchange water	150	-	150
Drum cleaning	20	-	20
Boiler feed water	400-600	120	-
Condensate recycle	-	60	-
Boiler blowdown	-	-	30
Total effluent	2620	1150	620

Table 3. Characteristic of effluent in various stages of starch processing (Nasr, Tawfik, okawara, & Suzuki, 2013) (Rausch, 2002) (Eckhoff & Watson, 2009).

Stage	pH	COD (mg/L)	BOD (mg/L)	Total solid (mg/L)	Total dissolved solid (mg/L)	SO_4 (mg/L)	Cl (mg/L)
Corn steep liquor	4.10-4.30	90000-113000	50000-80000	100000-120000	80000-100000	1500-3500	5000-8000
Gluten decanter	4.10-4.30	2800-3200	2000-2600	4700-5300	4400-4800	500-800	75-95
Starch decanter	4.5-6.2	3000-4500	1800-2700	3500-4500	1800-2200	300-800	25-65
Boiler blowdown water	11-11.5	300-700	10-30	6000-9000	-	-	-
Process cleaning water	4.5-6.0	2500-3500	1800-2300	3200-3900	-	-	-
Ion exchange water	4.10-4.30	800-1200	40-70	14000-18000	11000-15000	30-60	3800-4600

Understanding the biology of effluent treatment

The basic constituent of starch industry effluent contains large amount of carbohydrate and protein.

The fate of carbon and nitrogen in industrial wastewater is usually modulated through anaerobic and aerobic biological treatment processes (Chan *et al.*, 2009).

Mesophilic aerobic digestion of carbon and nitrogen offers 50% conversion of organic compounds by external physical co-factors and convert 50% into sludge which requires additional expenses prior to disposal. While anaerobic system is capable to reduce COD up to 90% and generates methane biogas, serves as a fuel (Abeynayaka and Visvanathan, 2011).

Fate of carbon pollutants

Anaerobic digestion of organic carbon is a multistep natural process mediated by several mixed microbial entity, predominantly archaea and methanogenic bacteria (Seghezzo *et al.*, 1998). In the first step complex organic material is broken down to simpler forms of sugar, amino acids and other volatile fatty acids like valeric, isovaleric, propionic, and butyric acids by fermentative anaerobic bacteria. Then this hydrolyzed products are converted to simple organic acids, CO_2 and hydrogen by acid formers. In the last step methanogenic bacteria converts volatile organic acids and derived alcohols to methane and carbon dioxide (Haandel A. V, 2007). The whole process is represented in Fig 2. In order to effective removal of nutrient, a dynamic equilibrium is required between acidogens, acetogens and methanogens. In an anaerobic digestion environment, syntrophic existence of nonmethanogenic obligate and facultative acidogens which produces hydrogen is utilized by strict anaerobic methanogens. In steady state a dynamic equilibrium exists between two groups (Gavrilescu, 2010). Anaerobic reactors like upflow anaerobic sludge bed reactor (UASBR), expanded granular sludge bed reactor (EGSBR), anaerobic filter bed reactor (AFBR), anaerobic fixed film moving bed bioreactor (AFFMBR), anaerobic digestion ultrafiltration (ADUF)are popular technologies to treat carbonaceous pollutants and to recover waste from water as methane gas. A comparison between different technologies and their efficiency is represented in Table 4.

Table 4. Performance of different reactors in treatment of starch industry effluent.

Reactor type	Application type	Operational pH	COD (mg/L)	HRT (h.)	Efficiency (%)	OLR (kg COD/m3/D)	Reference
EGSBR	Starch industry	6.9	2750	18.5	80	3.57	(Ersahin *et al.*, 2011)
AFBR	Lab. Reactor	6.7-7.3	2100-12900	10	96	27.5	(Zhang *et al.*, 2009)
ADUF	Starch industry	6.7-7.3	15000	124.8	97	2.9	(Ross *et al.*, 1992)
ADUF	Lab. Reactor	6.7-7.3	8000	38.4	90	5.0	(Ross *et al.*, 1992)
UASB	Lab. Reactor	6.8-7.9	45000	12	95.3	90	(Fang *et al.*, 2011)

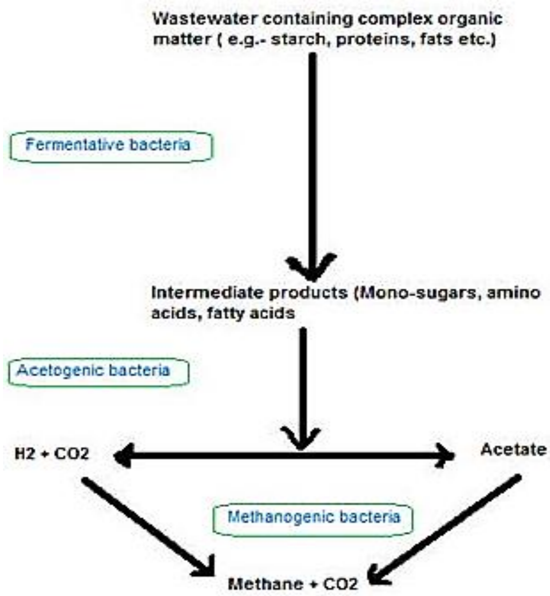

Fig. 2. Steps involved in anaerobic digestion process.

Fate of nitrogenous pollutants

Two basic approaches that have been widely adopted to control biological nitrogenous waste are; transformation to nitrogen gas of biological waste, and assimilation of nitrogen within microbial cell to increase cell mass. Assimilating nitrogen into cell mass is a widely practiced process throughout the world to remove nitrogenous waste from industrial & municipal effluents (Lu *et al.*, 2014). Nitrogen in effluent appears in a wide degree of complex forms, from protein to ammonia, ammonical, nitrate, nitrites, salt of ammonia, amino acids etc., where most of them contributes the formation of essential & non-essential amino acids for microbial cell after bioconversion (Bronk *et al.*, 2010).

Bioconversion of nitrogenous wastes mediates through various oxidation reduction states via nitrogen cycle, mediated by several group of bacteria, algae & few higher organisms.

Since, nitrogenous waste possess high risk of eutrophication, several technologies was developed and applied to treat nitrogen waste effectively from several industries. In the field of corn starch industry effluent treatment activated sludge process containing aerobic- anoxic system are very popular due to its short HRT and high load tolerance. Several other technologies are also applied in various industry like aerated lagoon, anammox process are also quite successful.

Understanding the technical aspects of different processes

The conventional method of nitrogen removal uses two stage treatment of effluent through nitrification and denitrification separately. Also recently a new pathway found for anaerobic ammonia oxidation by few group of chemolithoautotrophic bacteria from planctomycetes which are potentially capable of removing nitrogen pollutants from wastewater (Kuenen, 2008).

Nitrification-denitrification process

Biological degradation of ammonium biomolecules into nitrate via nitrite is known as nitrification. Nitrification, is a complex process, comprises a huge group of microbial flora that exist on a symbiotic syntrophic relationship within aerobic biological degradation process of wastewater.

The nitrification process is observed to be carried out mostly through two main group of chemo-litho-autotrophic proteobacteria; the ammonia oxidizing bacteria (Herrmann *et al.*, 2011), that belongs to β-subclass of proteobacteria and are monophyletic, and the nitrite oxidizing bacteria, belongs to α, γ, δ subclass of proteobacteria. Oxidation of ammonia in this process is mainly carried out by *Nitrosomonas* and *Nitrosococcus* groups of bacteria. Though a wide variety of other bacteria's are also capable of carrying out similar reaction, e.g. *Nitrosolobulas multiformis, Nitrosospira briensis* (Schmidt *et al.*, 2002). Formation of nitrite is mediated by *Nitrobactor & Nitrocystis* genera. e.g. *N. agilis, N. winogradsky, Nitrosococcu smobilis, Nitrospira gracilis* (Bitton, 2010).

$NH_4^+ + 1.5O_2 \rightarrow NO_2^- + H_2O + 2H^+$ + (240-350 kJ) (*Nitrosomonas*)

NO2- + 0.5 02 → NO3- + (65-90 kJ) (*Nitrobactor*)

The released energy in the above reaction is up taken by nitrifying organisms in presence of inorganic carbon source such as CO_2, HCO_3^- and carbonate compounds. The biomolecular pathway of the whole biological process of nitrification is represented in Fig 3.

Biological conversion of nitrate to nitrogen by bacterial degradation is known as denitrification process. This is oxygen free process utilizes nitrate as prime electron acceptor (Groffman *et al.*, 2009).

This pathway was observed to behave as both assimilative and dissimilative depending upon electron donor (represented in Fig 4). Under dissimilative anoxic condition nitrogen was observed to be released.

This process was observed to be carried out by a wide group of genera ranging from *Pseudomonas, Bacillus, Spirillum, Hyphomicrobium, Agrobacterium, Acinetobactor, Propionobacterium, Rhizobacterium, Cornebacterium, Cytophata, Thiobaccilus, Alcaligenes*etc. Nitrate serves as a final electron acceptor in dissimilatory nitrate reduction by fermentative bacteria, are mostly facultative anaerobes. Reduction of nitrite to gaseous form also depends on availability and nature of carbon source and the bacterial species involved(Yang *et al.*, 2012).

Several researchers reported an optimal C:N ratio of 3.0 to 6.0 for complete reduction to elemental nitrogen (Chiu and Chung, 2003) (Huiliñir *et al.*, 2011). The overall reaction is hypothesized as following-

$NO_3^- + 1/3\ CH_3OH \rightarrow NO_2^- + 1/3\ CO_2 + 2/3\ H_2O$

$NO_2^- + ½\ CH_3OH \rightarrow N_2 + ½\ CO_2 + ½\ H_2O + OH^-$

$NO_3^- + 5/6\ CH_3OH \rightarrow N_2 + 5/6\ CO_2 + 7/6\ H_2O + OH^-$

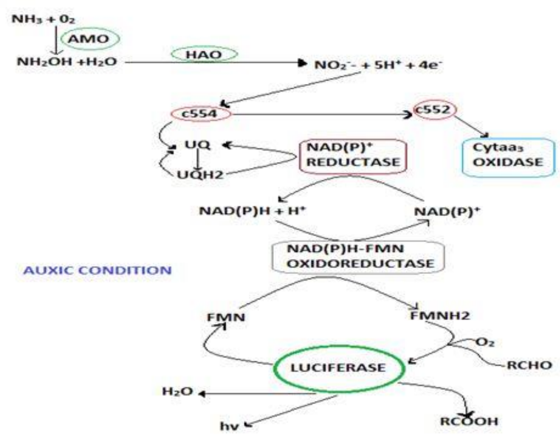

Fig. 3. Biological concept of nitrification process.

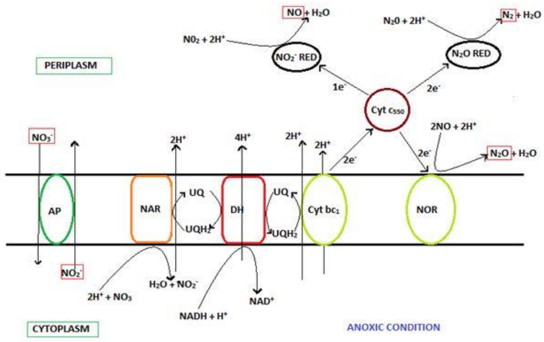

Fig. 4. Biological concept of de-nitrification process.

Anaerobic ammonium oxidation

Anaerobic ammonium oxidation, the most recent emphasized process, surprisingly changed our understanding of fate of nitrogen on biological removal process. Unlike conventional complex multistep process of nitrification and denitrification, this is a two-step process where oxidation of ammonia occurs in auxic condition and uptake of nitrite along with ammonia is consumed in the second step under anoxic condition, reducing overall energy utilization (Zhang *et al*., 2008) (Van de Graaf *et al*., 1995). Thus it is being considered as a cost efficient and environmental friendly technology to treat nitrogen contamination in waste water (Lackner *et al*., 2014) (Zhang and Liu, 2014). The unique metabolic pathway of anammox (represented in Fig 5) was first elaborated by Mulder based on thermodynamic calculation(Mulder, 1989). The anammox process utilizes nitrite as an electron acceptor and carbon dioxide for cell mass development under anoxic condition, thus contributes reduction of CO_2 also (Kartal *et al*., 2010).

Extensive studies were carried out in laboratory and in full scale to understand the process to utilizing it industrially on large scale. Though there is a conflict on the actual mechanism of the process and a huge research gap is still there to clearly decipher the process opening a new arena for extensive study on this field. The basic model of anammox reaction was stated as follows:

$5NH_4^+ + 3NO_3^- \rightarrow 4N_2 + 9H_2O + 2H^+$ ... (Van de Graaf *et al*., 1995)

$NH_4^+ + NO_2^- \rightarrow N_2 + 2H_2O$ ... (Strous *et al*., 1997)

$NH_4+ + 1.32NO_2^- + 0.066HCO_3^- + 0.13H+ \rightarrow 1.02N_2+ 0.26\ NO_3^- + 0.066CH_2O_{0.5}N_{0.15} + 2.03\ H_2O$ ... (Jetten *et al*., 2009).

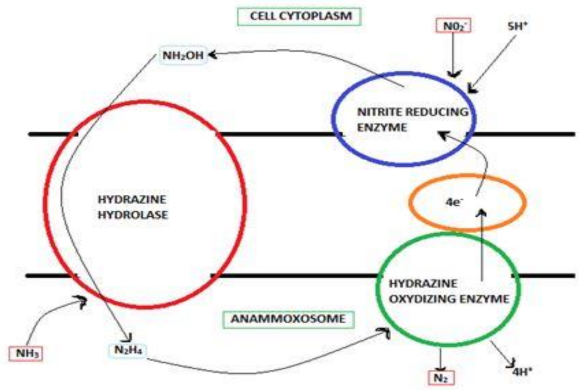

Fig. 5. Biological concept of ANAMMOX process.

Analysis of available technologies and their limitation in application for treating corn starch industry effluent

The basic treatment focus for corn starch industry is upon removing carbonaceous and nitrogenous nutrient where subsequent treatment by anaerobic bioreactor and auxic-anoxic treatment proved to be efficient. The conventional anaerobic reactors were designed non-specific to starch industry and thus faces several issues when treating acidic effluent like- operational limitation to pH 6.5-7.9, generation of huge amount of secondary waste sludge, high retention time. Similarly the most popular nitrification denitrification is also a high energy utilizing process due to recirculating design and vigorous aeration requirement to maintain dissolved oxygen (DO) level 5.6-6.0.

The newly developed ANAMMOX process overcomes above problems with nitrogen removal by less sludge production and complete anoxic operation. But the issue with this technology is also numeric and severe, like start-up of reactor requires longer time as the generation time of the bacteria is reported to be 11 days. There is no reliable data on onsite development of ANAMMOX sludge. Maximum reactor start-up was carried out using sludge from pilot reactor of 10L lab reactor from the laboratory of Rotterdam, Netherlands (Xiong *et al*., 2013) (Bagchi *et al*., 2012). Though several successful operation is still continued to treat different industrial wastewater by this process, but the microbiological process of the ANAMMOX activity is still not completely understood. The biochemical pathways are mostly hypothesized (Jetten *et al*., 2001). Also there is no specific culture technique or biochemical identification technique are still available to identify the process onsite. All the activities reported are based on critical genetic studies based on laboratory scale tests (Backman and Hulth, 2013). There is very few successful application of this technology reported for starch industry wastewater treatment in China (Ali *et al*., 2013) (Ni and Zhang, 2013).

Scope for new technologies for effective treatment of starch industries

Waste treatment is a low recovery process for any industry. So to overcome lacuna of treatment and to make the system more attractive and adaptable the treatment system must be specific according the characteristic of the effluent of concerned industry. Recent trend of industrial effluent treatment emphasizes to the possibility of carrying out methanogenesis under acidic condition. Methanogen cultivated from peat bog sludge shown to be highly active for utilization of bio-waste and gases like hydrogen and carbon dioxide (Kotsyurbenko *et al*., 2007). Duval and Goodwin (Duval and Goodwin, 2000) also reported that significant methanogenesis was observed under acidic pH. Several researcher investigated presence of low pH methanogen in peat bog where the pH goes down below pH 5 most of the time (Bhadra *et al*., 1984) (Jain and Mattiasson, 1998). Williams and Crawford (Williams and Crawford, 1984) (Williams and Crawford, 1985) reported active methanogenic activity up to pH 3.0-4.0. Several laboratory scale optimization study was conducted by different researchers to find out optimum condition for low pH methanogenesis, where most researchers has reported an optimal range between 4.5 to 5.0 (Maestrojuan and Boone, 1991) (Patel *et al*., 1993) (Bräuer *et al*., 2006). Recent research on low pH removal of corn starch industry effluent on laboratory scale also shown promising result (Neogi *et al*., 2016) by removing COD up to 69.78% at pH 5. More emphasis is required to find out the possibility of removing pH under acidic condition to make the effluent treatment system more cost effective, especially for industries like corn starch, potato starch, rice starch and other food processing industries which generates acidic effluent in bulk quantity.

Treatment of nitrogenous pollutant is a tedious job because of its highly diversified form of source starting from complex soluble and insoluble proteins, amino acids, nitrite, nitrate and ammoniacal salts (Padoley *et al*., 2008). Conventional effluent treatment system comprising aerobic-anoxic reactor operation is a cost effective procedure and increases overall HRT and waste sludge production. ANAMMOX process after successful application in China's corn starch industry definitely shows promising future. But for acceptable applicability confined research is required for identification and culture technique for anammox bacterial group for onsite development. Also there is an argument among researchers about its generation time, load tolerance and food to microorganism ratio (Hannides, 2014).

Zero discharge is also a principally appreciated approach for industrial manufacturing processes where reducing waste generation is the key target (Wang *et al*., 2015). Starch industries mostly generates huge amount of wastewater and waste biological sludge. The residue total suspended solid is a key barrier to recycle the water. The application of low cost coagulant or charcoal filtration along with reverse osmosis will prove to be an efficient and rapid technique for water reutilization and will help closing the loop (Chavalparit and Ongwandee, 2009). However, there is no sufficient data available on reutilization of wastewater internationally. Also the generated waste sludge usually dried and disposed as a cake of sent to boiler which further contribute to pollute land. Introduction to pyrolysis technology (Chen *et al.*, 2015) (Fonts *et al.*, 2012) using the generated methane from bioreactor will further help to generate energy efficient gas and fix the residue waste to non-leachable fixed waste which can be further used into construction material or for land filling purposes (Chen *et al.*, 2016).

Conclusion

Corn starch contributes 12% of total starch production and growing at 5.5% rate annually. Thus corn starch industry, now a days is an area of concern in term of global pollution contribution and control. The technologies available for treating starch industry waste water are not specific to the type and character of its huge amount of wastewater with high load, low pH and degradable solids. There is also lack of focus on reutilization of treated effluent and ensuring zero discharge in terms of water and waste sludge. Thus vigorous research emphasis is required on low pH bioreactor operation for carbonaceous effluent treatment along with nitrogenous pollutant removal by low cost denitrification with modification of available nitrification-denitrification or by anammox process. Introduction of secondary filtration-reverse osmosis to ensure generating reusable potable water is also a research gap that is present with today's available technology. Corn starch wastewater treatment thus leaves a wide area of opportunity for technological development in order to design cost efficient treatment system.

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There is no conflicts of interest between authors.

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