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Short communication

Treatment of Adsorbable Organic Halide (AOX) from pulp and paper industry wastewater using aerobic granules in pilot scale SBR

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ABSTRACT

Aerobic granulation of activated sludge was achieved in a pilot scale column type sequencing batch reactor (SBR) employed for the treatment of pulp and paper industry wastewater containing Adsorbable Organic Halide (AOX) in the range of 15–20 mg/L. Throughout the 780 days of operation the biomass trends to remain in between 7 and 8 g/L MLVSS and SVI reduced to 60–80 mL/g MLSS. After applying different selection pressure the average size of aerobic granule in the SBR was in the range of 2–4 mm. After achieving steady state conditions in the pilot plant, at an organic loading rateof 4.5 kg COD $m^{-3}day^{-1}$ at 6 h HRT the degradation of AOX was significant and the effluent AOX concentration of below 5 mg/L was achieved. The results achieved in the study exhibit the viability of formation of aerobic granules in pilot scale SBR and maintenance of long-term strength of aerobic granular sludge with a high reduction of AOX in pulp and paper industry wastewater.

1. Introduction

The pulp and paper industry is a water demanding production and ranks only third in the world, after the primary metals and the chemical industries, in terms of freshwater consumption [1]. Around 500 diverse chlorinated organic compounds have been identified including chloroform, chlorate, resin acids, chlorinated hydrocarbons, phenols, catechols, guaiacols, furans, dioxins, syringols, vanillins, etc [2,3]. The discharge of the Halogenated Organic Compounds in the environment is increasingly becoming a matter of concern due to their carcinogenic/toxic effects. Most of these compounds are adsorbable and in wastewater, these compounds are estimated in a group as "Adsorbable Organic Halides" (AOX).

The formation of these compounds is due to the use of chlorine and chlorinated compounds in the various industrial processes including the bleaching process in the pulp and paper industries. The pulp and paper industries also being one of the major consumers of chlorine and its compounds need main concern in the perspective of removal of AOX discharges in their effluents. The toxic effects of AOX (e.g. chlorophenols, guaiacols, furans and dioxins) range from carcinogenic and mutagenic effects to acute and chronic toxicity and can act as inhibitors of microorganism growth, [3–5]. Conventional wastewater treatment systems such as activated sludge process have been used for treatment of pulp and paper mill wastewater. The process has disadvantage of excess sludge production and fluctuations in removal efficiency for recalcitrant compounds [6]. Activated sludge process for treatment of pulp and paper mill wastewater have bulking problem, which in result causes poor settling of sludge and effluent have more suspended solids [7,8]. The use of chemicals such as chlorine, hydrogen peroxide, metal ions (Fe, Ca, Mg) are effective in getting better settling properties of sludge in paper mill wastewater treatment [9,10]. Moreover these chemical addition methods are not cost effective and they do not provide long term solutions for bulking problem. Conventional adsorption technique, post-treatment using a granular activated carbon (GAC) has been widely used for removal of AOX from pulp and paper mill wastewater. However GAC have to be regularly regenerated, because adsorption site are exhausted by pollutants after some time [11]. Osman et al. [5]. have used GAC in sequencing batch biofilm reactor (GAC-SBBR) to treat recycle paper mill wastewater and results showed high AOX removal efficiency by maintaining longest hydraulic retention time (HRT). The advantage of the biofilms in these systems save energy and less area is required, efficient to handle toxicity and can withheld high organic loading rate [12].

These days a new technology of aerobic granulation in column type Sequential Batch Reactors (SBR) have been widely explored based on modified activated sludge process. Due to the excellent settling capacity of aerobic granules, large area for a settling tank will not be required, which in turn reduces the wastewater treatment plant area by 80% [13]. Aerobic granules have unique structure and microorganisms that can withstand high loading rate as well as high toxicant concentration. Aerobic granulation can be considered as a special type of self-aggregation of microorganism, without any carrier medium for growth of

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biofilms. The immobilization of microbial cells into granule formation generally improved the ability of cells inside the granules to tolerate high toxicity concentration and achieve good structural properties and biodegradation performance. Sequential batch reactors have advantages of low operating costs, no secondary clarifier required, no recycling pumps required, and good control over filamentous microbial growth, tolerance to shock loads and denitrification during anoxic fill and settle phase [14]. Most of the lab scale and pilot scale studies were carried out for municipal wastewater treatment using aerobic granulation [15–18] and for synthetic wastes [13,19]. Very few studies have been carried out for development of aerobic granules on actual industrial wastewater like petrochemical wastewater [20], palm oil mill effluent (POME) [21], Brewery wastewater [22]. However, till date no work has been reported for the treatment of AOX compounds from pulp and paper industry using aerobic granulation technology. The present work carried out to evaluate the feasibility of a pilot scale SBR for the development of aerobic granules and to assess its performance for the biodegradation of AOX presents in the pulp and paper industry wastewater

2. Materials and methods

2.1. Reactor setup and operation

The Naini Tissue industry, Uttarakhand, India has been chosen for the study as it utilises agro based material and chlorine for bleaching purpose. The formation of AOX compounds occur when agro based materials are used as raw materials and chlorine is used for bleaching. The diameter of the pilot scale SBR was 600 mm and the effective height was 3000 mm resulting in total volume of 3.394 m^3 and height to diameter (H/D) ratio of 5:1 (Fig. 1). The Pilot Plant was seeded with the aerobic sludge of the aeration tank of the existing effluent treatment plant of the industry. Initially a 24 h cycle was maintained and was strictly followed. Later on the cycle of operation was reduced to 12 h, 8 h and 6 h. Programmable Logical Control (PLC) arrangements for the automation of the Pilot Plant SBR were used. Effluent was withdrawn from a port located at 40% height from the bottom resulting in volume exchange ratio of 60% (2.04 m^3).At maximum organic loading rate at HRT of 6 h the working capacity of pilot SBR was 8.16 m^3/day of wastewater.

2.2. Acclimatization and development process of aerobic granules

The pilot plant was seeded with the aerobic sludge of the aeration tank of the effluent treatment plant of the industry. The reactor was fed with the wastewater of the equalization tank. Diffused aeration was provided with the help of a blower and special types of diffusers were fitted at the bottom of the reactor which prevented the choking with sludge. Initially a 24 h cycle of operation was maintained which included fill react settle and decant timings of respectively. The samples were collected daily and were analysed for pH, alkalinity, Chemical Oxygen Demand (COD), Biochemical Oxygen Demand (BOD), Adsorbable Organic Halides (AOX). Provision of online measurement of dissolved oxygen was made using online HACH D.O. monitoring system. The sludge of the reactor was also analysed for its volatile content on a routine basis.

In the initial phase the reactor was fed with diluted pulp and paper industry wastewater with sewage (1 in 10) for proper acclimatization and growth of the microorganism. Thereafter the dilution was reduced to 1in 5, 1in 4 and 1in 3.sucessfully operating the pilot plant at reduced dilution for 220 days of operation wastewater having characteristics shown in Table 1 was fed to the reactor. The reactor was run on a 24 h cycle till a substantial removal of AOX, BOD and COD removal took place. The cycle of operation was reduced to 12, 8 and 6 h (on day 150, day 300 and day 475) once pseudo steady state conditions were achieved in each stage as shown in Table 2. The duration of each phase (fill, react, settle and decant) in individual cyclic operation is shown in Table 3. Initially settling time was kept at 30 min thereafter selection load was applied on day 150 as the settling time was reduced from 30 min to 5 min. This was done to retain the heavier aerobic sludge in the reactor which helps in formation of dense and compact aerobic granules. The reactor run was completed at 6 h of cycle after achieving a significant reduction in the values of AOX and COD and was operated for a total period of 780 days.

2.3. Analytical methods

Measurement of pH, biomass concentration, COD, BOD, Sludge Volume Index SVI were conducted in accordance with Standard Methods [23]. COD was measured using Closed Reflux method/Hach Reagents (DR5000 UV/Vis spectrophotometer, HACH, USA),pH was measured using HQ30d portable meter coupled with pH probe, HACH, USA. Continuous DO was measured using online HACH probe attached with PLC. Dissolved oxygen concentration (DO) was measured using HACH, USA HQ30d portable meter coupled with LBOD10101 probe. Samples were taken from the reactor at a predetermined time intervals for analysis.

Aerobic Granule was analysed using Cenisco binocular petrological microscope with SANYO digital camera. The photographs were analysed using image analysis (IA) system (Averz Software).The morphology of aerobic granules in stipulation to aspect ratio was analysed by using IA technique. Aspect ratio is the ratio between minor axis and major axis of ellipse corresponding to the granule (0 = line, 1 = circle). Granule strength is defined as an integrity coefficient (IC) (%) which is the residual volatile suspended solids (VSS) after sample has been agitated for 5 min at 200 rpm on a platform shaker to total VSS of the intact granules prior to agitation [24]. The stronger granules have higher ICs.

2.4. Adsorbable Organic Halide

The AOX measurements were done by Multi X 2500 halide analyzer (Jena, Germany). Nie et al. [25] described the basic method and process as follows: Effluent first passed through the activated carbon column, the organic chloride of the bleaching effluent then adsorbed by the activated carbon column. Sodium nitrate was used in washing the adsorbed inorganic chloride of the activated carbon column, and then the activated carbon column burned in the combustion furnace. Content of AOX calculated though the micro coulomb titration method [25,26].

2.5. Gas chromatography–mass spectrometry

GC (Gas chromatography) and GC/MS (Gas chromatography–mass spectrometry) analysis of influent and effluent samples were done in order to identify the number of products formed during aerobic biodegradation of pulp and paper mill wastewater. GC/MS equipped with a split/splitless injector and a Perkin Elmer Clarus 680, USA gas chromatograph interfaced with a Turbomass spectrometric (Perkin Elmer 600T, USA) mass spectrometric mass selective detector system were used. The samples were prepared and analysed under conditions as described previously [13]. The metabolic intermediates were derived from degradation of AOX identified by comparing their retention time (minutes) and mass spectra with that of the National Institute of Standard and Technology (NIST) library available.

2.6. Extracellular polymeric substances

The extracellular polymeric substances (EPS) of the flocculent sludge and aerobic granules were extracted using a heat extraction method as described earlier [27]. The carbohydrate content of the extract was analysed by the phenol-sulphuric method and expressed as the glucose equivalent, and its protein substance content were