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Treatment of rayon grade pulp drain effluent by upflow anaerobic fixed packed bed reactor (UAFPBR)

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Abstract: *The Rayon grade pulp (RGP) drain effluent of pulp and paper mill was studied to find out pollutant loading and its control measures by low cost and efficient treatment method. Upflow anaerobic fixed packed bed reactor (UAFPBR) with brick ballasts as packing material was used for this purpose. This was compared with conventional anaerobic treatment method. The digested slurry was taken as inoculum from the active cow dung biogas plant. After stabilization of the reactors the reduction in pollutant loading was found to be higher in UAFPBR than conventional anaerobic reactor (CAR). Hydraulic retention time (HRT) of 12 hr was optimum for the treatment of effluent when 74.5% COD and 81% BOD reduction was obtained. 30% inoculum concentration was best for the anaerobic treatment of RGP colour drain effluent. The maximum biogas production (*$1.37\ l\ l^{-1}$ *of effluent) was when the effluent was inoculated with 30% seeding material. Thus, UAFPBR system was very efficient in terms of BOD, COD, TSS and TDS removal from RGP drain of paper mills in ambient environmental conditions.*

Key words: *Bio-reactor, Pulp and paper mill, Rayon grade effluent, Anaerobic treatment*

Introduction

The increasing industrialization and its effluent discharges have accentuated the environmental problems to a large extent. Besides many other industries, the pulp and paper industries are also responsible for creating major water pollution (Adholeya *et al.,* 2006). These industries generate large volumes of wastewater for each metric ton of paper produced, depending upon the nature of the raw material, finished product and extent of water reuse (Singh *et al.,* 1996). Since the pulp produced corresponds to only approximately 40-45% of the original weight of the wood, the effluents are heavily loaded with organic matter. These effluents cause considerable damage to the receiving waters, if discharged untreated since they have high BOD (biochemical oxygen demand), COD (chemical oxygen demand), chlorinated compounds, suspended solids, fatty acids, tannins, resin acids, lignin and its derivatives (Escalante *et al*., 2005). The high polluting potential of pulp and paper industry wastewater can no longer be ignored. However, the major concern lies in the fact that even after more than 30 years of consistent efforts, a satisfactory treatment of these effluents still remains elusive (Ali and Sreekrishnan, 2001).

The anaerobic treatment of pulp and paper industry effluent has a number of advantages over aerobic treatment processes such as methane rich biogas, low nutrient requirement, low sludge production and overall low operation and maintenance cost (Webb, 1983; Singh *et al.,* 1994). A number of reports are available on the anaerobic treatment of effluent of various other industries such as sugar, distillery, dairy and Palm oil (Hall and Bank, 1993). However, only few studies have been made on anaerobic treatment of pulp and paper mill effluent (Dangcong and Qiting, 1993).

The anaerobic process is a biologically mediated process, is indigenous to nature and has a prominent role in wastewater treatment. The anaerobic process is said to be more efficient than aerobic one (Dayal and Upadhyay, 1999). There have been many significant advances in the implementation of anaerobic digestion processes with respect to industrial and domestic wastewater treatment (Stronach *et al*., 1986).

The anaerobic lagoons are traditionally used for this treatment. However, the major disadvantages with the above are slow growth rate of microorganisms including methanogens and large sized land requirement, which restrict the use of anaerobic lagoons. Fortunately, they attach themselves readily to the surfaces or to other bacteria, and this characteristic has made it possible to develop reactors as are known today. All the advanced anaerobic treatment systems are based on same kind of microbial immobilization principle in order to retain as much viable biomass as possible (Tare and Javed, 1999).

According to Ali and Sreekrishnan (2001) the processes employed in pulping as well as pulp processing (including pulp bleaching) are so diverse that the composition of the resulting wastewater are very different and no single process or combination of processes can apply to all for treatment.

In the pulp and paper industries, various effluent drains are generated depending upon the types of processes used. The seasonal characteristics of RGP colour drain effluent were described

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recently by Malaviya and Rathore (2007). Mean values of temperature, pH, chlorides and total phenols of the effluent were reported to be below, whereas colour, BOD_5, COD and lignin concentrations were above the minimum national standards (MINAS, 1985).

The RGP colour drain effluent of pulp and paper mill was selected for two reasons. First, it contributes nearly 58% of the total wastewater generation from the mill. The effluent discharge from this RGP colour drain is about 6000-7000 $m^3 day^{-1}$. Secondly, the pH of this effluent varies from 8.0 to 9.5 and hence this range of pH is more suitable for anaerobic treatment and biogas production (Patel and Madamwar, 2000; Sambo *et al*., 1995).

Considering the facts as mentioned above, an alternative treatment method is suggested for the pulp and paper mill effluent. This experiment was conducted to find out the optimum percentage of inoculum required for anaerobic digestion in the reactor in terms of biogas production.

Materials and Methods

The study was conducted on RGP colour drain effluent released from Century Pulp and Paper Mill Ltd., Lalkua, Nainital, Uttaranchal (India). The effluent was collected for pollutant loading analysis from main RGP colour drain stream from inside the factory.

Inoculum optimization: Digested slurry from cow dung based biogas plant (Fixed-dome plant with a brick reinforced, moulded dome) was collected from Livestock Research Centre of G B Pant University of Agriculture and Technology, Pantnagar. This slurry was filtered with muslin cloth and active bacterial suspension was brought to laboratory as seeding material. The inoculum obtained from digested slurry (active bacterial suspension) was added to the effluent to make different concentrations (15, 30, 45 and 60%) of inoculum in the effluent. The total volume of effluent and inoculum was kept 300 ml by putting different ratio of effluent and inoculum in 600 ml plastic bottles. The bottles were having an outlet for the gas release. After filling with effluent and inoculum, mouth of each bottle was sealed by synthetic adhesive.

Rubber tubing was placed on the outlet of the bottle and it was closed with pinch cork to create anaerobic conditions and make the bottle airtight. One set of different concentrations of inoculum bottles was kept in incubator at 35 ± 2^{o}C and other set of these were kept in ambient environmental conditions to find out anaerobic digestion in terms of biogas production in both conditions. The biogas produced in bottles was measured by water displacement method.

Reactor operation: A schematic diagram of the experimental reactor set up is shown in Fig. 1. For designing laboratory scale reactor, cylindrical shaped containers made up of iron sheets were used. The volume of one reactor was 20 liter with 35 cm height and 24 cm diameter. All the reactors were provided an inlet at the bottom for the upflow of effluent and an outlet at the height of 27.5 cm from bottom for the discharge of treated effluent. For the collection of biogas from reactor an outlet was provided at the top.

For packing of the upflow anaerobic fixed packed bed reactors, brick ballasts of 3 to 4 cm diameter were used up to half of the reactor height. This reactor was basically designed for slime development on fixed packed bed materials. Here, maximum anaerobic biomass growth occurs due to larger and rougher surface area in this reactor as compared to the conventional anaerobic treatment system. It is operated in a vertical upflow mode, so that, effluent gets maximum contact with slime and sludge while moving upwards in the reactor whereas, conventional anaerobic reactors were used without any packing material. After packing of the reactor it was fed with 10 liter RGP colour drain effluent that was inoculated with 30% seed culture. The reactor openings were sealed with M-seal to ensure anaerobic conditions in the reactor. The narrow opening at the top of the reactor was connected to water displacement bottle with a plastic pipe. The reactor was stabilized for 30 days in ambient environmental conditions. Thus, two reactors were run in parallel with one of them acting as control.

After substrate stabilization in both the types of reactors, experiments were carried out to determine optimum HRT of the reactor for pollutant reduction. The hydraulic retention time is the theoretical amount of time in which waste flows or remains in a reactor completely undisturbed or in quiescent conditions for settling of a large percentage of suspended particles and is in contact with slime in the reactor. For calculating this HRT, 2 liter of effluent was fed into the reactors through the inlet provided at the bottom for upflow. A hydraulic retention-time of 6, 12 and 24 hr was given to the effluent in each reactor for a period of 3 days. Finally, a performance evaluation was done by comparing the pollutant removal efficiency of conventional anaerobic reactor and Upflow anaerobic fixed packed bed reactor at the above given hydraulic retention time in ambient environmental conditions. For the study of pollutant loading of the effluent parameters such as COD, BOD, TSS (total suspended solids) and TDS (total dissolved solids) were determined by the methods as described in APHA (2005). The soluble COD was analysed after centrifuging the samples for 10 minutes at 5000 rpm.

Duncan's multiple range test (SPSS Inc., version 10.0) used for data analysis to assess the significance of quantitative changes in biogas production and effluent characteristics after treatments.

Results and Discussion

Pulp and paper industry wastewater has been traditionally treated by aerobic means. In the last few years, extensive studies and an increasing number of full-scale applications have been demonstrated for high rate anaerobic processes. Use of these methods proved beneficial to reduce the load of pulp and paper industry wastewater (Rintala and Lepisto, 1992). The operation stability and satisfactory performance of an Upflow anaerobic sludge blanket reactor, is to a great extent, dependent on the kind of sludge cultivated in it. A good sludge should have good settleability and high methanogenic activity (Wu *et al.*, 1987). The sludge from a cowdung biogas plant is the best source of inoculum with enhanced microbial activities for substrate utilization and biogas yield (Boopathy, 1987). In the present study seed (inoculum) was taken from an operating cowdung biogas plant. The experiment was carried out for the stabilization of UAFPBR and CAR with 30% inoculum. A comparative study of UAFPBR and CAR was made for biogas production and pollutant removal efficiency. For the packing of UAFPBR small brick ballasts were used. The brick stones have rough surface for better growth of slime on it. The stabilization period of 30 days was given to the reactors and marked reduction in all the measured pollutant parameters were found.

Inoculum optimization: In the sample having 15% inoculum concentration, it was observed that the average total biogas production was 375 ml with 300 ml seeded effluent (1.25 l l^{-1}) in a time period of 30 days at controlled laboratory conditions (35 ± 2°C) in an incubator. Whereas the average biogas production from 15% seeded effluent, under ambient environmental conditions was 174.3 ml from 300 ml effluent in the time period of 30 days (581 ml l^{-1}). The total biogas production in ambient environmental conditions was 53.6% less than the total production observed in controlled laboratory conditions.

In the sample having 30% inoculum concentration, it was observed that the average total biogas production was 413 ml with 300 ml seeded effluent (1.37 l l^{-1}) in a time period of 30 days at controlled laboratory conditions (35 ± 2°C). Meanwhile, the average production in ambient environmental conditions was only 202 ml with 300 ml effluent (676.4 ml l^{-1}). Hence, the total biogas production in ambient environmental conditions was 51% less than the total biogas production observed in controlled laboratory conditions.

In the bottles having 45% inoculum concentration the average biogas production was 308 ml from 300 ml effluent (1.02 l l^{-1}) in a time period of 30 days in controlled laboratory conditions (35 ± 2°C). On the other hand the total biogas production under ambient environmental conditions was 157.7ml from 300 ml seeded effluent (525.9 ml l^{-1}). This biogas production was around 50% less than the total biogas production under controlled laboratory conditions.

The observations with 60% inoculum were also made in similar manner with incubated samples at 35 ± 2°C in laboratory conditions. On an average total biogas production of 222 ml was obtained from 300 ml of seeded effluent (737.8 ml l^{-1}).

Under the ambient environmental conditions total biogas production of 112 ml was obtained from 300 ml seeded effluent i.e. equal to 374 ml l^{-1}. The total biogas production in this case was 50% less than that produced under incubated conditions.

A control, containing effluent without seeding, was also run with the above-mentioned experiments in both controlled laboratory conditions (35 ± 2°C) and ambient environmental conditions. Almost negligible amount of gas production was found with this sample in both conditions. The average of daily biogas production in incubated (35 ± 2°C) and ambient environmental conditions of the 30 days experiments are given in Fig. 2,3 respectively. The total biogas produced during 30 days of experiment with above mentioned inoculum percentage in laboratory and ambient environmental conditions are shown in Fig. 4.

The ambient environmental temperature during the study period varied a lot. The maximum temperature varied from 16.4 to 28.5°C and minimum temperature varied from 3.4 to 14.5°C.

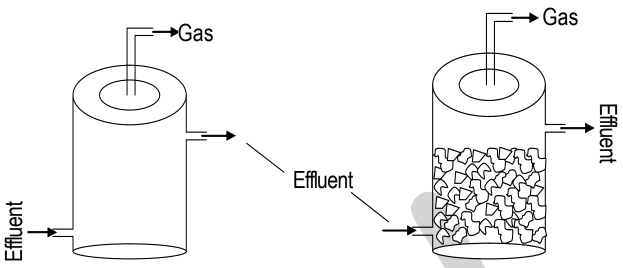

Fig. 1: Schematic diagram of experimental rack for effluent generation from various streams of rayon grade pulp unit

In the present investigation, it was found that in both ambient environmental conditions and controlled laboratory condition, 30% inoculum level showed maximum biogas production in comparison to other concentration of inoculum *i.e.* 15, 45 and 60% as shown in Fig. 3. It was also found that in all concentration of inoculum the biogas production in ambient environmental conditions was nearly around 50% less of biogas produced in controlled laboratory condition at 35 ± 2°C. This shows that the temperature and other environmental factors have marked effect on biogas production. However, similar reports were given by Chawla (1986) that at temperature of 15°C the biogas production was less than 50% of that produced at 35°C.

During the period of experiment that fall in winter season, thin and moderate fog was prevalent in most of the day in the sky. This fog reduces the solar radiations reaching the earth surface and subsequently affect the biogas production due to fluctuation in temperature between 3.4 to 28.5°C. Therefore, it may be concluded that 30% inoculum is the optimum concentration for the start-up of reactors under ambient environmental conditions.

The biogas production was noted on daily basis by water displacement method during the period of stabilization of reactors. The biogas production in UAFPBR started on 4th day of commencement of experiment with 5 ml l^{-1}. It gradually increased to 100 ml l^{-1} on 23rd day. Following this, a constant rate of 100 ml l^{-1} per day was noticed up to 30th day of the experiment. Thus, on an average total biogas production of 15.7 l was measured in 30 days from 10 l of seeded effluent.

Similarly, in conventional anaerobic reactor (CAR) the biogas production started on the 5th day of start-up of the experiment. The initial biogas production in CAR was 5 ml l^{-1}. The production rate gradually increased up to 61 ml l^{-1} per day upto 27th day of the experiment and remained constant up to 30th day. On an average the total biogas production by CAR was 10 l from 10 l seeded effluent.

The daily biogas production in UAFPBR and CAR during 30 days is shown in Fig. 1. This figure reveals that the total biogas production is around 50% higher in UAFPBR than in CAR. The maximum temperature during the experimental period varied from 30.0 to 38.5°C and minimum varied from 12 to 20°C as shown in Fig. 1 and during these temperatures, as days of stabilization of reactor increases, the biogas production also increases up to 26 days from the start-up of the experiment.

Reactor operation: The performance of both UAFPBR and CAR were determined in terms of reduction efficiency of pollutants from

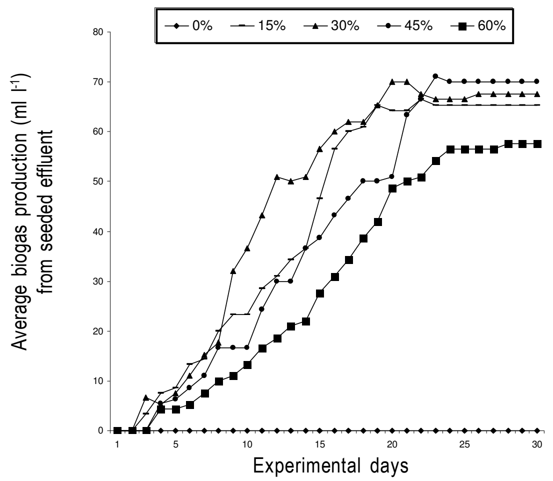

Fig. 2: Average biogas production with different concentration of inoculum at 35 + 2°C in controlled laboratory conditions

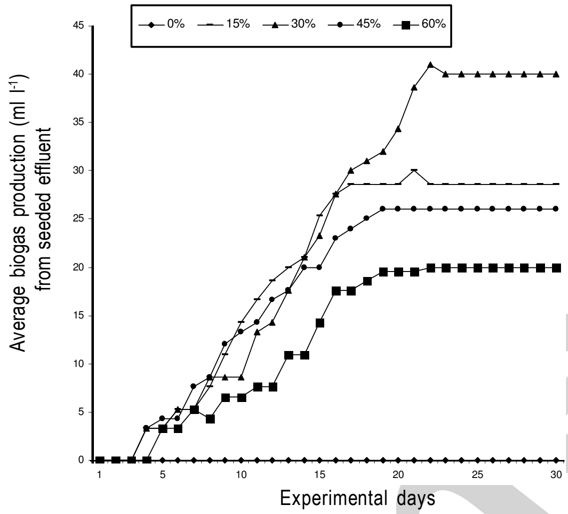

Fig. 3: Average biogas production with different concentration of inoculum in ambient environmental conditions

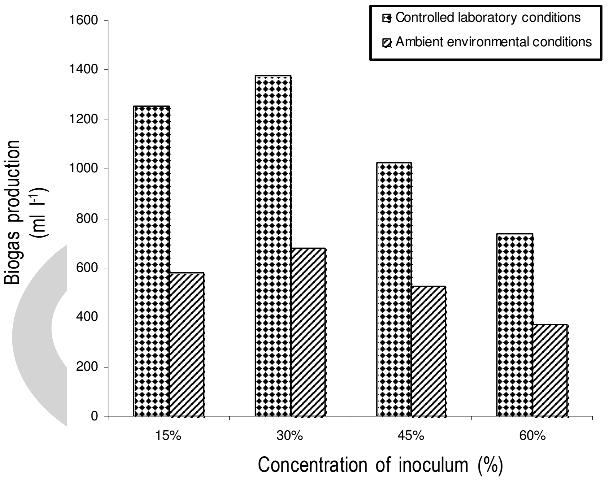

Fig. 4: Total biogas production (ml l^{-1}) with different inoculum concentration over a period of 30 days (Bars with different letter significantly differ according to Duncan's multiple range test at p<0.01)

treated effluent. A fresh amount of two liter of untreated effluent (without inoculum) was added to all the reactors through inlet provided at the bottom of the reactor for upflow movement of effluent in the reactors which were already kept for stabilization for a period of 30 days. Further, experiment was carried out to determine percentage reduction of pollutant loading at different given HRT.

On 1st, 2nd and 3rd day after 30 days stabilization of reactors the pollutant loading of effluent was analyzed after providing a hydraulic retention time of 6, 12 and 24 hr up to a period of 3 days and it was compared with the initial concentration of pollutant loading of the effluent as given in Fig. 5.

At 6 hr hydraulic retention time (HRT) the COD reduction in UAFPBR was found to be 64.7% as compared to 52.9% in CAR. The BOD removal efficiency was 73.4% in UAFPBR and 57.6% in CAR. Similarly a reduction of 34.3% was found for TDS in UAFPBR and 25.3% in CAR. In spite of higher reduction of all the pollutants in the UAFPBR, it was also observed that the TSS removal efficiency in UAFPBR was 27.8% and in CAR was 51.5%.

A COD reduction efficiency of 74.5% was measured in UAFPBR and 62.2% in CAR at 12 hr HRT. A reduction of 81.0% was found in BOD value in UAFPBR and the corresponding value in CAR was 66.8%. Similarly, the reduction of 62.7 and 52.0% was observed in TSS and TDS respectively in UAFPBR. A reduction of 71.6 and 51.0% respectively was observed for TSS and TDS in CAR. Following the same trend as 6 hr HRT, the TSS removal efficiency was still higher in CAR as compared to UAFPBR.

At 24 hr HRT the COD reduction in UAFPBR and CAR was 74.7 and 62.3% respectively. Reduction of 81.3% was found in BOD value in UAFPBR and 67.4% in CAR. The TSS and TDS removal efficiency in UAFPBR was 56.4 and 52.0% respectively while the corresponding values in CAR was 60.2 and 51.6% respectively.

The COD and BOD percentage reduction efficiency measured from Upflow anaerobic fixed packed bed reactor at different given hydraulic retention time of 6, 12 and 24 hr are compared in Fig. 2 and 3 in same ambient environmental conditions. The results obtained in both the reactors at a different HRT show that upflow anaerobic fixed packed bed reactor has better efficiency for pollutant load reduction.

Bishnoi *et al*. (2006) reported the biodegradation of pulp and paper mill effluent using anaerobic followed by aerobic digestion. Using a continuous stirred tank reactor (CSTR) for anaerobic digestion of black liquor, these authors reported a maximum methane production was found up to 430 ml day^{-1}. According to Pathe *et al*. (1995) upflow stationary fixed film (USFF) reactor reaches the steady stage after a period of 30 days. In the present study after stabilization of reactors, reduction in studied parameters of effluent *i.e.*, COD, BOD, TDS and TSS was higher in upflow anaerobic fixed packed bed reactor than the conventional anaerobic reactors. Chian and DeWalle (1977) has also reported that removal efficiency in fixed bed reactor was higher than anaerobic digesters operated at same volumetric loading for high strength acidic wastewater.

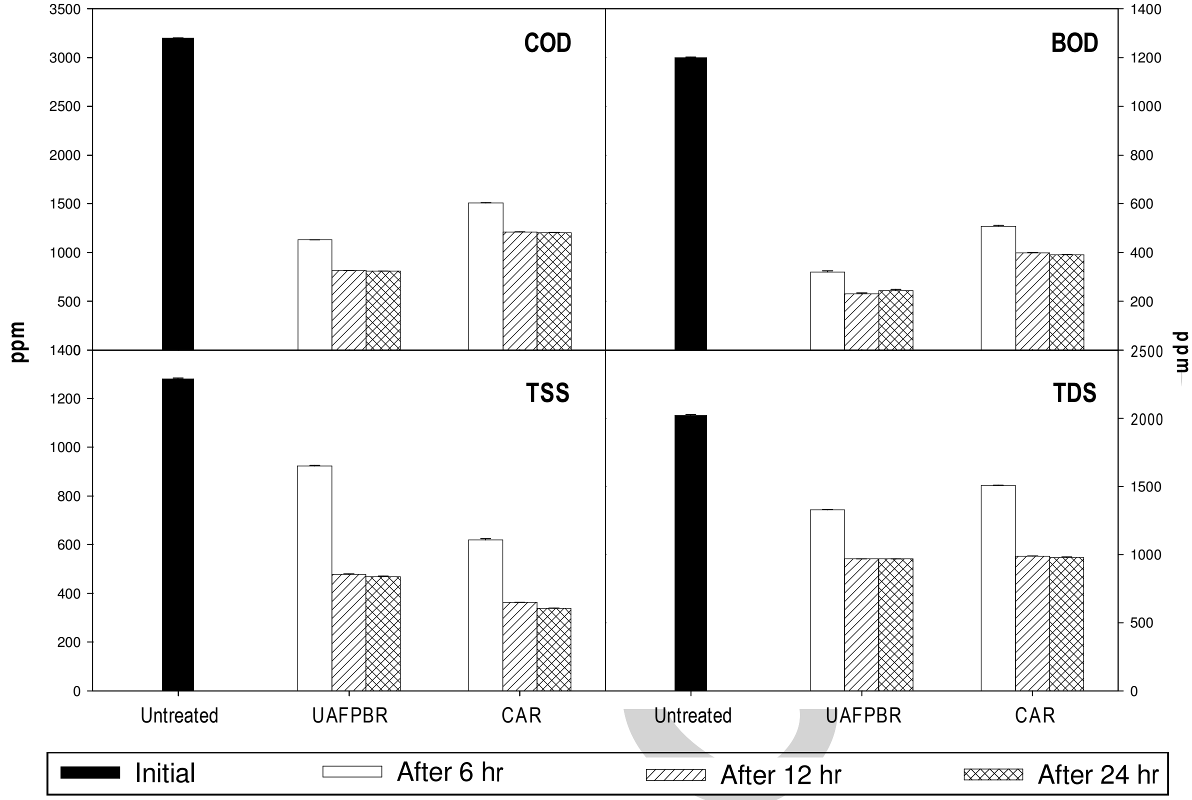

Fig. 5: Changes in COD, BOD, TSS and TDS of Rayon grade paper mill effluent (Bars with different letter significantly differ with in each parameter according to Duncan's multiple range test at p<0.01)

During the stabilization period of this experiment 1.57 l biogas per liter of effluent was produced in upflow anaerobic fixed packed bed reactor as compared to 1 l l^{-1} in CAR. The COD during this period was reduced to 410 mg l^{-1} from an initial value of 3200 mg l^{-1}. The total reduction in COD was 2790 mg l^{-1}. According to Basu and Leclerc (1975) the biogas produced from anaerobic filter contains 60-65% methane. Based on this study the methane production by the removal of 2790 mg l^{-1} of COD was 0.94 l of effluent (60% of 1.57 liter). This result shows that for reduction of 1 g COD the methane production was 330 ml. The obtained result corresponds to the reported theoretical value of 380 ml methane production from the removal of 1 g of COD at standard temperature and pressure (McCarty, 1964).

In conventional anaerobic reactor the total biogas production during the period of stabilization was 1 l l^{-1}. The COD in this reactor was reduced to 1000 mg l^{-1} from the initial value of 3200 mg l^{-1}. Therefore, the total COD removal in this reactor was 2200 mg l^{-1}. Based on the study conducted by Basu and Leclerc (1975) the methane produced in this type of reactor will be 0.6 l l^{-1} (60% of the total biogas production). According to this 0.27 l of methane was produced in the reduction of 1 g COD in conventional anaerobic reactor. This shows that there is lesser biogas production in conventional anaerobic reactor than in the upflow anaerobic fixed packed bed reactor.

The experiment carried out for optimization of hydraulic retention time, reveals that HRT of 12 hr is best suited for RGP colour drain effluent having a COD of 3200 mg l^{-1}. Generally a HRT of 22-24 hr is given for the wastewater of high COD loading ranging from 7000 to 10,000 mg l^{-1} (Li *et al.*, 1982). But Berg (1984) has suggested that to obtain high loading with relatively dilute waste the hydraulic retention time has to be short.

The reduction in the values of COD, BOD, total dissolved solids was higher in the upflow anaerobic fixed packed bed reactor than the conventional anaerobic reactor. But the reduction in the value of total suspended solids was found to be higher in conventional anaerobic reactor. This lesser reduction in UAFPBR could be attributed to the use of brick ballasts and their fine particles as packing material in it.

However, the reduction of total dissolved solids was higher in the UAFPBR as compared to conventional anaerobic reactor. This is of greater significance because dissolved solids contribute towards much of the COD of the effluent. On an average upflow anaerobic fixed packed bed reactor is more efficient than conventional anaerobic reactor at the same HRT and same volumetric loading.

Therefore, the experimental results show that the fixed packed bed reactor gave better performance because it consists of packing media as shown in Fig. 1. This packing material provides more rough surface area to which microorganisms get easily attached and form a thick layer of slime.

The reason for the development of alternative treatment method for treatment of RGP colour drain effluent was to get maximum pollutant removal efficiency and methane recovery. In the present investigation COD value was reduced from 3200 to 800 ml l^{-1} at 12 hr hydraulic retention time after stabilization of reactor. The rate of effluent generation from RGP colour drain of pulp and paper mill selected for this study is around 6000-7000 m^3 day^{-1}. For this large amount of effluent the COD reduction by UAFPBR will be 14.4 kg COD m^{-3} day^{-1}. According to theoretical value, 1 kg COD reduction produces 330 l of methane (McCarty, 1964). Thus, for the reduction of 14.4 kg COD the amount of methane produced will be 4752.02 l. According to Chawla (1986), the calorific valve of methane is 33.2-39.6 J cm^{-3}. Therefore, from 475.2 cm^3 of methane 15776.6 Joule energy can be produced during treatment of this effluent and it will be equivalent to 1095.5 J kg^{-1} of COD reduction. Therefore, apart from higher pollutant removal efficiency this process is also energy generating rather than energy utilizing.

According to the study made by Speece (1985), 1.1 kwh of electric power is consumed for the removal of 1 kg COD by aerobic treatment method. Therefore, for 14.4 kg COD m^{-3} day^{-1} reduction, the electric power requirement will be 15.84 kwh and this energy can be saved by using UAFPBR in which energy requirement is almost nil and pollutant removal efficiency is better than conventional anaerobic treatment method.

To conclude, the results presented here demonstrate that the removal efficiency of upflow anaerobic fixed packed bed reactor for pollutants such as BOD, COD, TSS and TDS were very satisfactory in ambient environmental conditions. In ambient environmental conditions the maximum temperature varies from 30.0 to 38.5°C. To further increase the effluent treatment efficiency of UAFPBR and output of methane gas, a research is required to adjust C:N ratio, volatile fatty acids and measurement of methane concentration in biogas production. This treatment system will be more economical as compared to aerobic system because of almost negligible power requirement and less operation and maintenance cost.

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