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Biomethanation of Dye Industry Wastewater

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Abstract:

Dyestuff sector is one of the core chemical industries in India. Maharashtra and Gujarat account for 90% of dyestuff production in India due to the availability of raw materials and dominance of textile industry in these regions. During industrial processing up to 40 % of the used dyestuffs are released in to the process water. The untreated effluents released from the dyeing units cause a major threat to the environment. Direct discharge of dye effluents causes formation of toxic aromatic amines in receiving media. The majority of colour removal techniques work either by concentrating the colour into the sludge or by partial or complete breakdown of the coloured molecules. Although a variety of effective physical and chemical treatment methods are commercially available, most of them are either expensive, not adaptable to a wide range of dyes, or do not completely solve the problem of complete decolourization of dye containing industrial effluents. Biodegradation have been explored as a method of biological treatment of dye. Hence in a present study attempts were made to biologically treat dye industry wastewater by biomethanation. Anaerobic digestion of 25% dye industry wastewater was studied under various environmental conditions and observed that COD reduction of 88.88%.BOD reduction of 68.06% giving 73.57% colour removal. In addition, anaerobic treatment gave valuable fuel in the form of methane which adds to the revenue of the industry. The sludge obtained after anaerobic treatments posses good manurial value and hence adds to revenue of industry.

Keywords: Dye effluent, anaerobic digestion, biomethanation, decolourization, manurial value

1.0 Introduction:

Dyestuff sector is one of the core chemical industries in India. India accounts for around 5% of the global output. It is also the second highest export segment in chemical industry. Maharashtra and Gujarat account for 90% of dyestuff production in India due to the availability of raw materials and dominance of textile industry in these regions. Thus organized sector contributes 65% while small scale sector contributes 35% in overall dyestuff production in the country. The major users of dyes are textiles, paper, plastics, printing ink and foodstuffs. The textiles sector consumes around 80% of the total production due to high demand for polyester and cotton, globally (Khan, 2003). Dyes, however are more difficult to treat because of their synthetic origin and mainly complex aromatic molecular structure, such structures are often constructed to resist fading on exposure to sweat, soap, water, light or oxidizing agents and this renders them more stable and less amenable to biodegradation(Banat *et al.,* 1996). Still these dyes can be removed from environment by microbial methods of decolourization and degradation which are cost effective (Verma and Madamwar, 2003; Asgher *et al.*, 2007).Dye wastewater usually consists of a number of contaminants including acids, bases, dissolved solids, toxic compounds and colour. Colour is the most noticeable contaminant even at very low concentration and it needs to be removed or decolourized before the wastewater can be discharged (Chu, 2001). Wastewaters of the industries have following characteristics: High levels of COD and BOD, High acidity, deep colour of different shades, high levels of chlorides. During industrial processing up to 40 % of the used dyestuffs are released in to the process water (Vaidya and Date, 1982; Faraco *et al.,* 2009). The untreated effluents released from the dyeing units cause a major threat to the environment. Direct discharge of dye effluents causes formation of toxic aromatic amines under anaerobic condition in receiving media (Murugesan and Kalaichelvam, 2003). Some dyes are reported to be carcinogenic causing cerebral abnormalities in foetuses and skeletal abnormalities (Murugesan and Kalaichelvam, 2003; Rajendran and Gunasekaran ,

2006) Azo dyes have been linked to human bladder, spllenic sarcomas, hepatocarcinomas and nuclear abnormalities in experimental animal and to chromosomal aberrations in mammalian cells(Rajendran and Gunasekaran, 2006).

Treatment of wastewater containing dyes and its decolourization is very difficult due to wide ranges of pH, salt concentration and chemicals. Although a variety of effective physical and chemical treatment methods are commercially available, most of them are either expensive, not adaptable to a wide range of dyes, or do not completely solve the problem of complete decolourization of dye containing industrial effluents. Biological methods are currently viewed as effective, specific, less energy intensive and environmentally benign since they result in partial or complete bioconversion of organic pollutants to stable non toxic end products (Kuhad *et al*., 2004).

For decolourization of azo dyes a wide range of organisms are able to reduce azo compounds under anaerobic conditions.
Two mechanisms for the decolourization of azo dyes under anaerobic condition in bacterial system have been proposed (McMullan *et al*., 2001; Pearce *et al*.,2003; Doble and Kumar, 2005).
1) Direct transfer of electrons to azo dyes as terminal electron acceptor via enzyme during bacterial catabolism, connected to ATP generation.
2) It involves a free reduction of azo dyes by the end products of bacterial catabolism, not linked to ATP generation i.e. reduction of azo bond by reduced inorganic compounds such as Fe^{+2} or H_2S, that are formed as the end product of certain anaerobic bacterial metabolic reaction (McMullan *et al*., 2001; Stolz, 2001; Kuhad *et al*., 2004; Doble and Kumar, 2005).

A major advantage of anaerobic system apart from the decolourization of soluble dyes is the production of biogas. Biogas can be resulted to provide heat and power and well reduce energy costs (Manilal *et al*., 1990; Robinson *et al*., 2001; Keharia and Madamwar, 2003; Kuhad *et al*., 2004; Rajendran and Gunasekaran,2006). Additional advantages are no aeration requirement, less sludge generation and methane gas production. Kanekar and Kelkar (1995) treated anaerobically phenol bearing dyestuff wastewater in laboratory scale upflow anaerobic filter made up of PVC pipe and packed with polypropylene rings. The filters were initially stabilized on cattle dung and the feed was gradually replaced by the dyestuff wastewater at 10 days HRT at 35^{o}C for 78 days. The anaerobic treatment results in 91-94% removal of phenol with production of biogas containing 67% methane. Talarposhti *et al*. (2001) reported colour condition using two phase anaerobic packed bed reactor. This can remove colour up to 90% from a mixed cationic dye containing 1000mg/L of dye. Colour removal efficiency falls as the influent dye concentration increased but rises with increased HRT and increased organic loading. Bras *et al*. (2001) studied behaviour of methanogenic and mixed bacterial cultures on the colour removal in batch systems of a commercial azo dye. C.I. Acid orange 7. They observed that in mixed culture colour removal was faster than in methanogenic culture in which an adaptation time seemed to be required. Dye degradation occurred for both groups of bacteria when co-substrate in form of addition of electron donors such as sucrose, glucose and acetate stimulate the reductive cleavage of azo bonds giving more than 94% decolourization. At higher dye concentration more that 300 mg/L, there is an apparent inhibition effects in COD removal rates. Manu and Chaudhari (2002) decolorized commercially important azo dyes orange II and Reactive Black 3 HN, under anaerobic condition in wastewater. Colour removal of more than 99% was achieved in both dye containing reactors. COD removal up to 92-94% was achieved. Hence in a present study attempts were made to anaerobically treat the wastewater.

2 Material and Methods:

2.1 Collection of Dye Industry Wastewater:

Wastewater of Spectrum Dyes and Chemicals Pvt Ltd, Surat, India was selected for the present study. The industry manufactures reactive, azo, acid, direct and disperse dyes. Throughout the year the industry manufactures approximately 69 different types of dyes and the quantity of the products depend upon the market trend. Hence the effluent in the present study contains diverse types of dyes. Samples of industrial streams of effluent were collected separately in 50 L plastic carboys. All samples were immediately transported to the laboratory, tested and mixed in equal proportion to be used as composite waste and preserved at 4°C in refrigerator and were used as and when required during the course of study (APHA, 1992) and Trivedy and Goel (1984).

2.2 Analysis of Dye Industry Wastewater:

Effluent samples collected were analyzed for their physicochemical characteristics. The examination was carried out by the methods described in APHA (1992) and Trivedy and Goel (1984).

2.3 Anaerobic Treatment of Dye Industry Effluent:

To carry out studies on anaerobic treatment of dye industry effluent floating dome batch anaerobic digester of KVIC design (Gober Gas, 1975) was used. The effluent was fed after pH adjustment to neutral with lime followed by settling (Nandy and Kaul, 2001; Borja *et al.,* 1995). As process of biomethanation works optimally with a C: N ratio around 30:1(Goel, 2003; Manilal *et al.,* 1990), cattle dung (C: N ratio 20:1) (Goel, 2003) was mixed in the proportion of 75:25 with wastewater under study so as to make slurry.

Several studies have reported that suitable co-substrate and alkalinity in a bioreactor are necessary for colour removal. The electrons required for decolourization are provided by an electron donating carbon source such as glucose (Isk and Sponza, 2005). Bicarbonate alkalinity was used to provide favourable conditions for conversion of substrate to methane. Hence 0.1% of sodium bicarbonate was added in the fed influent (Isk and Sponza, 2005; Bras *et al.,* 2005; Bras *et al.,* 2001). Molasses was used as a source of co- substrate as reported by various researchers as primary substrate which increases methane producing ability of organisms (Jadhav *et al.,* 2007; Zee and Villaverde, 2005; Kapdan 2005; Assadi *et al.,* 2003).As the effluent was deficient in phosphorous, 0.05% super phosphate was added. Since the effluent was highly acidic in nature and to prevent corrosion of the bioreactors, stainless steel bioreactors were used as reported by Isk and Sponza(2004).The reactors were initially run on cattle dung slurry (Cattle dung: water 1:1) having 25% total solids (Goel,2003) at 30 days Hydraulic Retention Time (HRT) at 28±2°C (ambient temperature). After the reactors were stabilized for methanogenesis as evidenced by methane content of 50% in the biogas generated, they were charged with admixture of cattle dung slurry prepared in dye wastewater. Three cycles of 30 days HRT were run and then used for optimization of parameters by using 2 L anaerobic fed batch reactors. The total gas production was monitored by water displacement method. (Ghangrekar *et al.,* 2005; Nandy and Kaul, 2001; Borja *et al.,* 1995; Soto *et al.,*1993)Process of biogas production from organic matter is a complex microbiological phenomenon and hence gets affected by different environmental factors particularly pH and temperature.

2.3.1 Optimization Parameters of Biomethanation:

2.3.1.1 Effect of pH:

The efficiency of the process is restricted to a normal range of pH, 6.5 to 7.5. A drop in pH below 6.0 results in the failure of digester characterized by less or no methane production (Ranade and Gadre, 1988). Hence the reactors were fed with 75:25 admixture of cattle dung and dye wastewater of different pH as 6.5, 7.0, 7.5 and 8. The pH was adjusted with lime. Daily samples were withdrawn, centrifuged at 5000 rpm until a clear supernatant was obtained. Analyses of the clear supernatant were carried out for COD, BOD and pH (Kapdan 2005). Amount of biogas produced was measured by water displacement method. The pH value showing maximum production of methane was taken as an optimum.

2.3.1.2 Effect of Temperature:

Anaerobic digestion takes place efficiently at ambient temperature of 25 to 40°C, since majority of anaerobic bacteria responsible for the process have temperature optima of 30 to 40°C. With fall in temperature their metabolic activities retard and hence the process becomes slow (Ranade and Gadre, 1988). Temperatures selected for the study were ambient temperature (28±2°C), 37°C and 40°C.The incubator was used to get 37°C and 40°C. The temperature at which maximum production of methane obtained was taken as an optimum temperature.

2.3.1.3 Effect of Hydraulic Retention Time:

As the time required for acclimatization of organisms in degradation of dye wastewater varies based on composition of the wastewater, the reactors were operated at 25, 30, 35 and 40 days HRT. According to HRT, every reactor was fed daily with the calculated amount as mentioned by Hawkes and Hawkes (1987). The HRT at which maximum volume of methane was produced was taken as an optimum.

2.3.1.4 Scale Up Of the Process:

Using optimized parameters at 2 L level (pH 7.5, Temperature 28±2°C and HRT 30 days),the process was scaled up to 10 L digester capacity and then final scale up was done to 50 L and the digesters were fed with admixture o f 75:25 cattle dung and dye industry effluent and outlet was analyzed everyday for COD, BOD, pH colour and amount of biogas. N, P and K of sludge levels were also studied. Biogas produced at all parameters was measured by water displacement method and was collected in small vaccine bottles and analyzed by gas chromatography for detection of % methane (Toshniwal Instruments, India equipped with thermal conductivity detector and stainless steel Porapak Q column, with carrier gas hydrogen at 10 ml/min and column temperature was room temperature)(Kanekar and Kelkar, 1995).

2.3.1.5 Colour removal efficiency

Under optimized conditions, colour removal was studied by sepctophotometrically.

3.0 Results and Discussion:

3.1 Analysis of Dye Industry Wastewater:

Throughout the year the industry manufactures approximately 69 different types of dyes and the quantity of the products depend upon the market trend. Hence the effluent in the present study contains diverse types of dyes. The composition of dye industry effluent (DIE) was found to vary significantly. This may be due to the batch production of the dye, variation in the recovery process and the raw material used (Sarnaik and Kanekar, 1995). Talarposhti *et al.* (2001) reported that the wastewater characteristics from the dye house are highly variable from day to day and even hour to hour depending up on the type of dye. Our results are in agreement with the facts stated above. The industry generates 950m^3/day of wastewater. The effluent shows variation in pH depending on the type of dye manufactured and mostly ranging from 1 to 2 pH and high in salt concentration such as chlorides, COD & BOD (Table 1). This is in agreement with the Mou (1991) and Sarnaik and Kanekar (1995). Dye industry wastewater characteristics reported by Bhatia (2002) show high levels of BOD & COD, high acidity, high TDS, deep colour of different shades, high levels of chlorides& sulphates, presence of phenolic compound, presence of heavy metals, presence of oil & grease. These characteristics support our result.

3.2 Anaerobic Treatment of the Effluent:

Many azo dyes constituting the largest dye group may be decomposed into potential carcinogenic amines under anaerobic conditions in the environment. Colour removal from wastewater is often more important than to the removal of soluble colourless organic substances which usually contribute the major fraction of COD & BOD (Wong and Yu, 1999). Among industrial wastewater, dye wastewater from textile and dyestuff industry is one of the most difficult to treat. This is because dyes usually have a synthetic origin & complex aromatic molecular structure which makes them more stable and more difficult to be biodegraded (Husseiny, 2008). Biological, they methods being cheap and simple to use have been the main focus of recent studies on dye decolourization and dégradation (Banat *et al.*, 1996). Azo dye toxicity to anaerobic biomass has been reported in some of the reactor studies. This toxicity was associated to high dye concentration, the presence of heavy metals (metal complex dyes) and presence of non hydrolyzed reactive groups of reactive dyes (Zee and Villverde, 2005). Therefore, the digester was acclimatized by the proportion of 75:25 proportions to cattle dung and dye wastewater respectively (Jang *et al*, 2007; Nandy and Kaul , 2001; Kanekar and Kelkar, 1995). Three cycles of 30 days hydraulic retentions time (HRT) of 75:25admixture were run and then used for optimization of parameters by using 2L laboratory scale fed batch reactors.

3.3 Optimization of Parameters of Biomethanation:

3.3.1 Effect of pH:

Amongst all selected pH, at 7.5 pH 88.88% and 68.06% reductions in COD and BOD were observed respectively. There was gradual increase in production of biogas from 100 ml to 355ml. pH of the outlet of the digester was found to be 8.0 Kapdan(2005) reported COD removal of 82% by using Upflow Anaerobic Sludge Blanket (UASB) for decolourization of textile dyestuff. Isk and Sponza (2005) noted that generally during the anaerobic process a reduction in COD of up to 60-70% can also be achieved. Jadhav *et al.* (2010) reported COD reduction of 78% of textile effluent treated with bacterial consortium. O'Neill *et al.* (1999) described COD removal of 70-95 % in anaerobic-aerobic system in a simulated textile effluent containing modified starch. At all pH, pH of outlet was always in the alkaline range. The pH rise up to 7.7-7.9 was also reported by Keharia *et al.* (2004). Sandhya *et al.* (2005) also reported shift in pH of the medium to alkaline range. This may be due to the formation of ammonia from aromatic amines. No methane was detected at pH 6.5. Percentage methane was 56.61, 58.53 and 58.34, respectively at pH 7, 7.5 and 8.0. Hence pH 7.5 was selected as the optimum for maximum COD and BOD removal as well as for maximum biogas production. (Fig a, b, c and d)

3.3.2 Effect of Temperature:

One of the most important parameters for anaerobic treatment of wastewater is operating temperature. There was an increase in COD removal from ambient temperature (28±2°C) to

37°C. It was observed that 88.88% and 89.04 % reduction in COD and 68.06 and 69.42 % BOD reductions were observed at ambient and 37°C, respectively. At 40°C, 48.47 % reduction in COD was observed. At 40°C BOD reduction of only 9.90% was noted. Biogas production at ambient temperature was found to be maximum reaching 355 ml. At 37°C and 40°C, biogas production of 220 ml was observed. There was not much difference observed in the results at both ambient and 37°C. Collins *et al.*(2005) reported that the mesophilic range is traditionally used since it is generally thought that maintaining a high temperature is uneconomical for many wastewaters where as degradation within psychrophilic range is too slow. At all temperatures, pH of outlet was always in the alkaline range.(Fig e, f and g). Hence anaerobic treatment was carried out at ambient temperature (28±2°C).

3.3.3 Effect of Hydraulic Retention Time

At 30& 35 days HRT 88.88 %, 68.06 % and 55.76% and 6.6 % of COD and BOD reductions, respectively were observed. The pH of the outlet under the HRTs selected was found to be alkaline. Amount of biogas produced at the selected HRT in sequence is 80,430,305 and 50ml on an average per day. Percentage of methane produced at both HRTs was found to be 56.61 % and 57.35 % at 30 & 35 days, respectively. (Fig h, i, j and k).

3.3.4 Scale Up of Anaerobic Process: Using optimized parameters at 2 L digester viz pH 7.5, HRT 30 days and (28±2°C) temperature, the batch digester was fed with 75: 25 of admixture of cattle dung & dye industry effluent. The process was scaled up to 10 lit digester capacity and then final scale up was done to 50 L digester. At 10 L scale up, 25 % effluent showed 85.24%, 68.00 and 70.25% reduction in COD, BOD and colour removal. Scale up studies of 50 litre level showed reductions of 88.88%, 68.06% in COD and BOD, respectively. Maximum 73.57% colour removal was observed (Fig l).

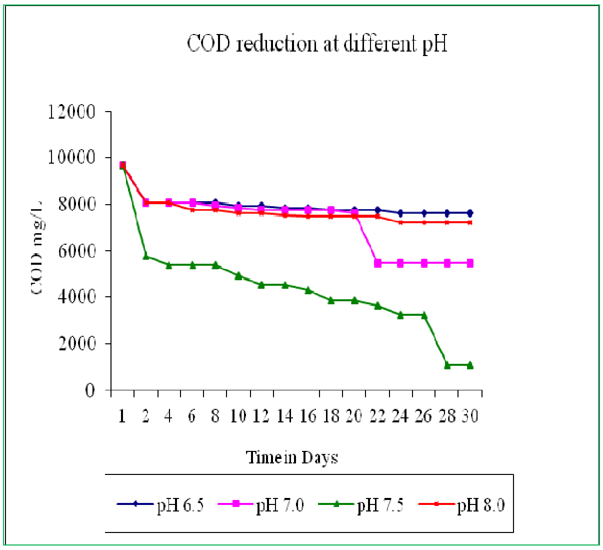

Fig a: COD reduction at different pH

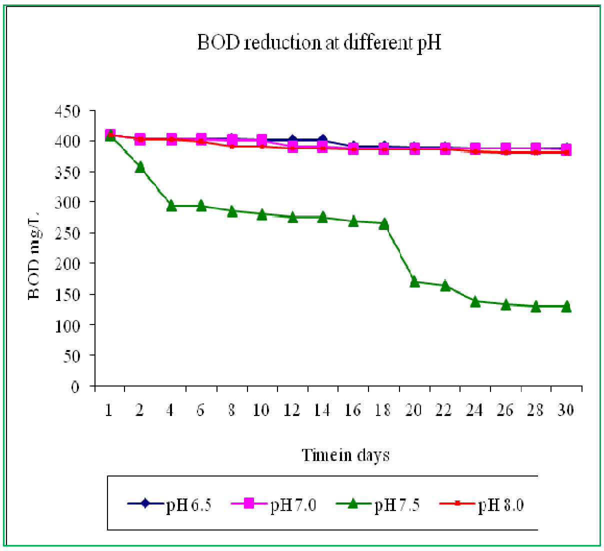

Fig b: BOD reduction at different pH

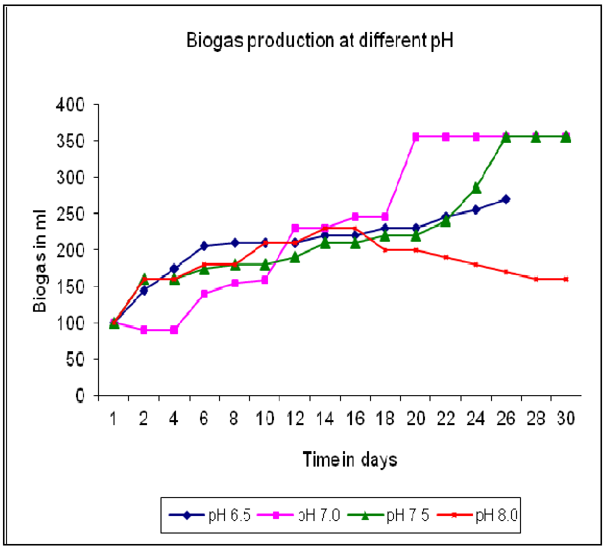

Fig c: Biogas production at different pH

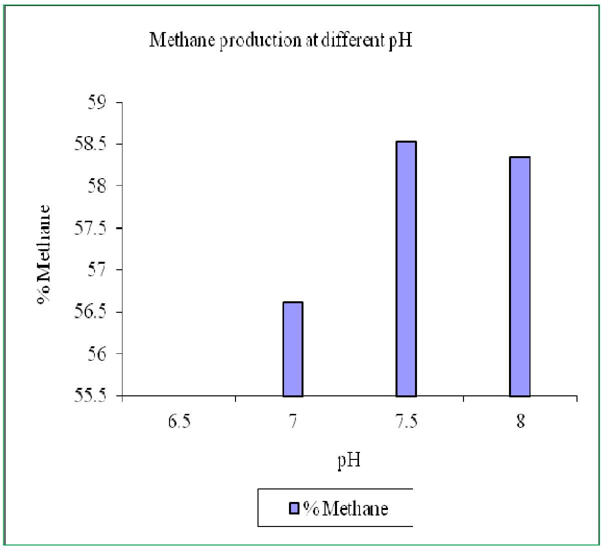

Fig d: % Methane production at different pH

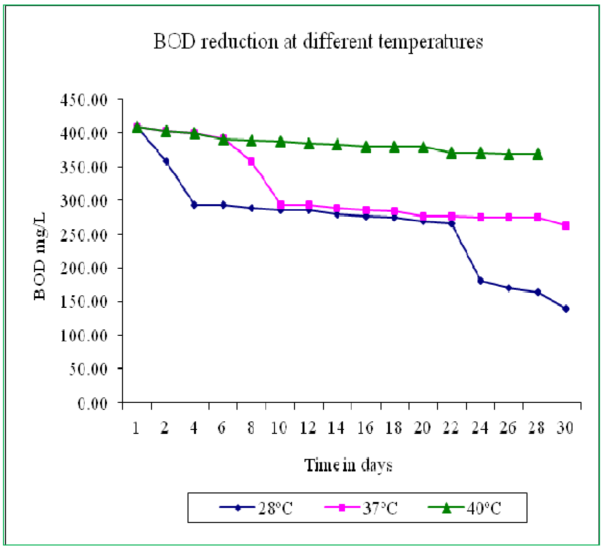

Fig f: BOD reduction at different temperature

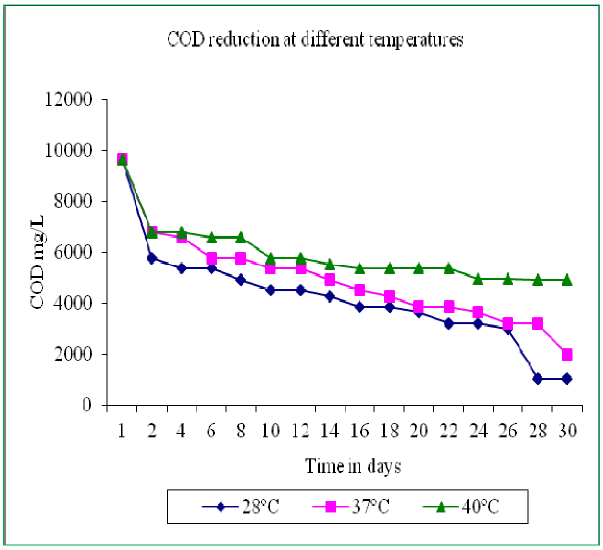

Fig e: COD reduction at different temperature

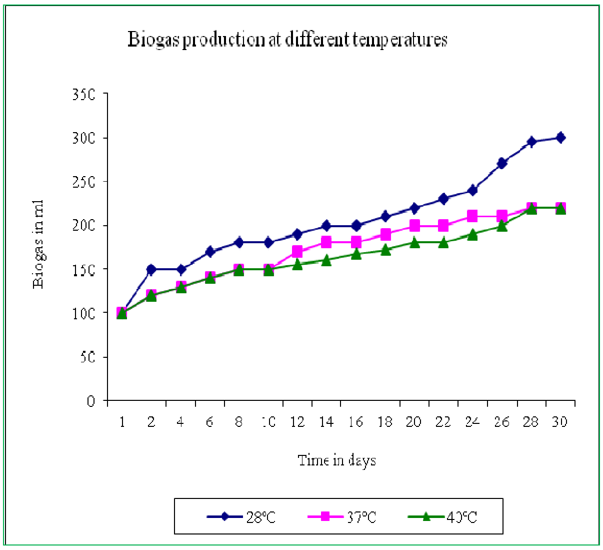

Fig g: Biogas production at different temperatures

Table 1 Physicochemical characteristics of composite DIE

Parameter	Values
COD	9000mg/L
BOD	480mg/L
pH	1.2-1.8
Colour	Dark brown to wine red
Chlorides	13206mg/L

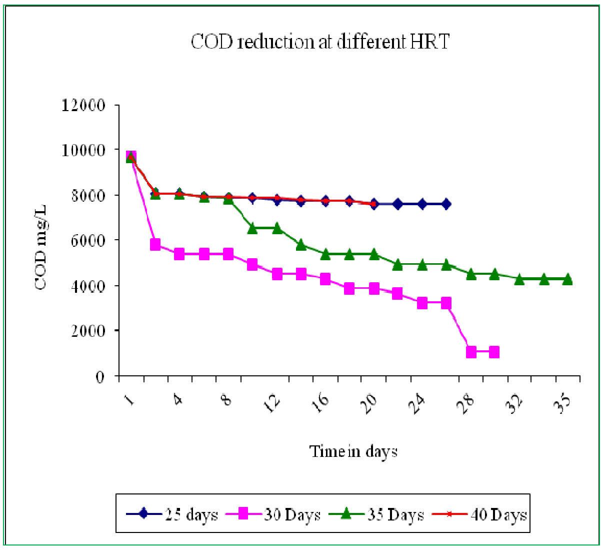

Fig h: COD reduction at different HRT

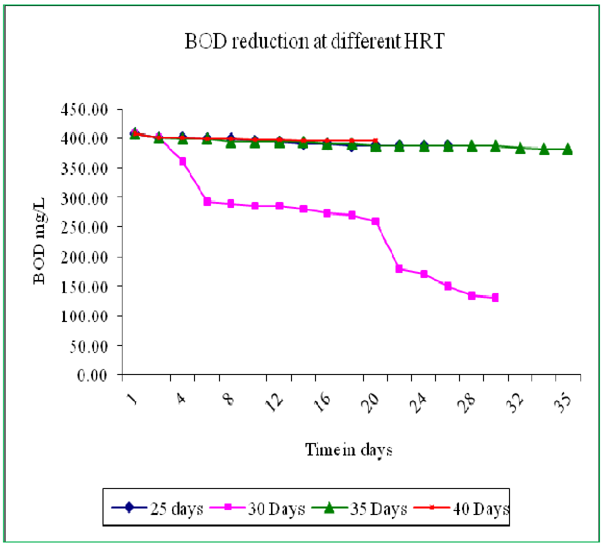

Fig i: BOD reduction at different HRT

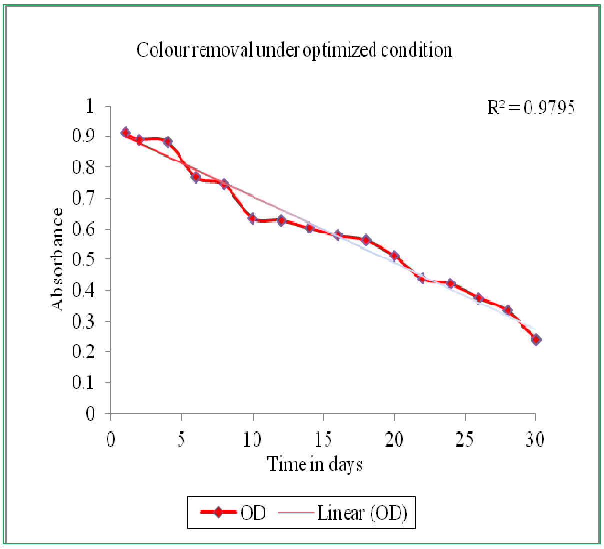

Fig l: Colour removal of 25% effluent feed under optimized conditions

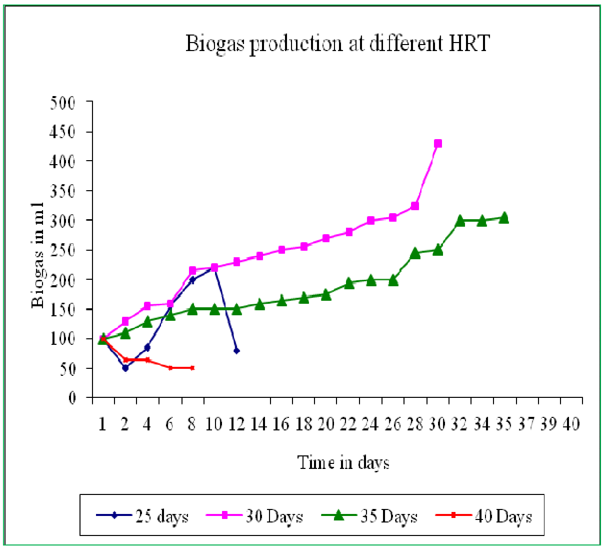

Fig j: Biogas production at different HRT

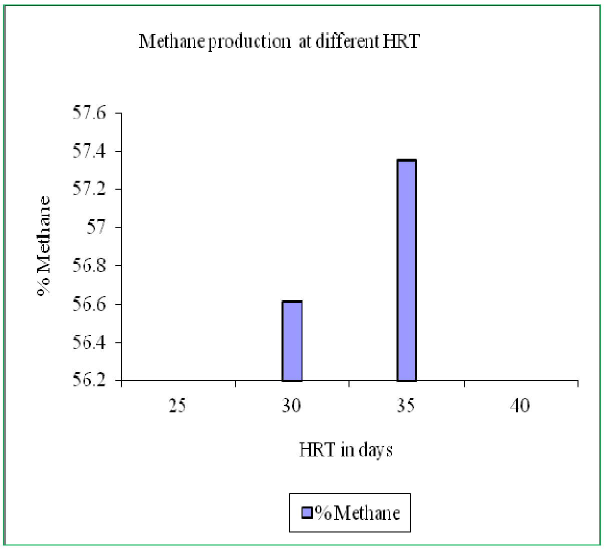

Fig k: % Methane production at different HRT

Gnanapragasam *et al.*(2010) used UASB with anaerobic digester sludge treating textile dye wastewater and noted 96% removal of COD and 93.3% removal of colour, at 30% recycled treated wastewater. In above study, slightly less removal of COD and colour was observed which may be due to complex nature of the dye industry wastewater. Baeta *et al.* (2011) carried out anaerobic degradation of the azo dye blue HFRL in bench scale UASB operated at ambient temperature and reported low COD removal of approximately 35%. In a present study much higher COD removal than above report was observed. Anaerobic sludge was found to contain 47mg/L of N, 104 mg/L of P and 240mg/L of K which is higher than the untreated effluent indicating the use of the sludge as manure. Methane produced can be used as fuel. This adds to revenue of the industry. Thus anaerobic treatment of dye industry wastewater by biomethanation was found to be cost effective.

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