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An efficient biotreatment process for polyvinyl alcohol containing textile wastewater

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Abstract

The aim of this work was to optimize the biodegradation of polyvinyl alcohol (PVA) containing actual textile wastewater for a sustainable treatment solution. The isolated microbial consortia of effective PVA degrader namely *Candida* Sp. and *Pseudomonas* Sp., which were responsible for symbiotic degradation of chemical oxidation demand (COD) and PVA from desizing wastewater. In the process optimization, the maximum aeration was essential to achieve a high degradation rate, where as stirring enhances further degradation and foam control. Batch experiments concluded with the need of 16 lpm/l and 150 rpm of air and stirring speed respectively for high rate of COD and PVA degradation. Optimized process leads to 2 days of hydraulic retention time (HRT) with 85–90% PVA degradation. Continuous study also confirmed above treatment process optimization with 85.02% of COD and 90.3% of PVA degradation of effluent with 2 days HRT. This study gives environment friendly and cost effective solution for PVA containing textile wastewater treatment.

Key words: biodegradation, environment, sustainable treatment, symbiotic degradation

INTRODUCTION

Sizing and desizing are the key textile processes where the use of polyvinyl alcohol (PVA) is unavoidable due to its functional and economically beneficial characters. Textile processing is the major source of industrial water pollution across the world and sizing – desizing operations account for nearly 30% of the water consumed in a textile plant (Chen *et al*. 2013a). The main problem resulting from the desizing step is the high load of chemical oxidation demand (COD) found in the polymer-containing effluent (Bechtold *et al*. 2004). These washed polymers include starch, PVA, carboxymethyl cellulose and acrylates. PVA and starch are the major contributors of organic load with 0.01 and 0.67 BOD to COD ratio respectively (Matsumura *et al*. 1993; Yu *et al*. 1996; Rajasimman & Karthikeyan 2007). China, Japan and the United States continue to be the world's largest producers and consumers of PVA. It is predicted that its consumption will increase at an overall rate of about 3.6% annually during 2012–2017 (Inoguchi and chin, 2010). Though PVA is not toxic, it has a great surface activity and can form large amounts of foam in the water. This foam affects the oxygen content in water body, thereby inhibiting or even undermine the respiratory activity of aquatic organisms (Zhang *et al*. 2005). The desize pollution streams containing synthetic PVA are hence a threat to the environment (Hao & Zhao 1994; Porter 1998). Although considerable efforts have been made to replace PVA, it has not been possible to develop a warp sizing chemical that can match the sizing performance of PVA and at the same time be cost-effective and biodegradable in effluent treatment plants (Chen *et al*. 2013b).

A number of studies explain the improvement in the biodegradability of PVA using advanced oxidation processes (Jing *et al*. 2008; Wu *et al*. 2008; Oh *et al*. 2009), but their application and treatment economy needs to be considered for the process viability. Similar attempts to study membrane

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filtration (Lee *et al*. 1999; Mo *et al*. 2008), vacuum flash evaporation (VFE) (Gupta 2009), ultrafiltration (UF) (Porter 1998; Altin 2008) has been made of PVA recycle and reuse after the treatment. But high initial investment cost and operation – maintenance cost makes these options unattractive to textile industries for implementation and use.

Lower COD textile effluents can be treated easily and the water can be reused after any tertiary treatment (He *et al*. 2013). But for treatment of high effluent COD of 10,000 to 20,000 mg/l with 0.5 to 1% PVA concentration need well performing microbial consortia with optimized process. There is no any systematic process research available which will lead towards process optimization of PVA biodegradation and it can be viable for commercialization. The objective of the present study was to isolate effective microorganisms for treatment of actual PVA containing textile wastewater and to optimize the feasible process parameters, such as aeration and stirring to reduce hydraulic retention time (HRT) for high COD and PVA degradation.

MATERIAL AND METHODS

Wastewater sample collection and analysis

An actual visit was arranged to loom processing site of a textile industry at Kolhapur MIDC, Maharashtra, India. Gathered information about sizing mixture use for textile processing, which contains Starch – Solite; 74%, Binder-Elevenoll T-66 (Dow Chemicals), PVA 90-50, PS-83, UCF – 4; 24% and Softener – RLS-21, Glisofin; 1%. 100 liter of PVA containing desizing wastewater sample and 10 liter of dye containing a separate stream of wastewater sample from textile dying and washing unit were collected. Both collected samples were analyzed for COD, BOD, % PVA, pH, Chlorides, SO_4, HCO_3 and Na.

Isolation of capable microbial strains

Different fungal and bacterial strains were isolated from samples from textile factory, as well as from our factory premises. Selective microbial isolation was done on agar media 1 (AM1) by serial dilution. Composition of AM1; PVA 1%, sodium nitrate 3.0 mg/l, potassium chloride 0.5 mg/l, magnesium sulfate heptahydrate 0.5 mg/l, Iron (II) sulfate heptahydrate 0.01 mg/l, di-potassium hydrogen phosphate 1.0 mg/l, Agar 15.0 mg/l and adjust final pH 7.3 $\pm$ 0.3. Isolated strains were grown on a nutrient broth for enrichment and stored at -197 °C.

Media preparation and inoculation for selection of efficient PVA degrader

Synthetic PVA was used to prepare the varying concentration of carbon source; 0.5, 1 and 2% PVA with some other nutrients (sodium nitrate 3.0 mg/l, potassium chloride 0.5 mg/l, magnesium sulfate heptahydrate 0.5 mg/l, Iron (II) sulfate heptahydrate 0.01 mg/l, di-potassium hydrogen phosphate 1.0 mg/l). Solubilization and heat sterilization of media containing different concentration of PVA were done at 110 °C after adjusting the final pH of all flasks to 7.3. Four isolated effective strains and one mixed (containing all cultures) were inoculated in 0.5, 1 and 2% PVA concentration flask and incubated at room temperature with constant shaking at 150 rpm.

Optimization of batch treatment process

Actual PVA containing desizing wastewater was used for optimization of the treatment process. All the experiments were carried out at room temperature. Three types of treatments were used, namely aeration, stirring and aeration with simultaneous stirring. Each experiment was done with

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one liter PVA containing actual textile wastewater. Stirring at 150 rpm and air was 8 l/min provided in this experiment. Actual PVA containing wastewater was used after heat sterilization at 110 °C, by adjusting initial pH 5.4 with 1N HCl and 1N NaOH. All six sets were inoculated with 5% respective microbial inoculums of TS1, TS2, TS3, TS4, and TSM (TS Mixed). PVA degradation was analyzed at after 5 days by Finaly Method (Finley 1961). Microbial Gram's staining was done for TS mixed reactor culture to observe acclimatized microbial flora.

Isolation and identification of acclimatized microflora

Serially diluted acclimatized culture of TSM was streaked on sterilized agar plates with 1% PVA as the only carbon source and incubated at 35 °C for 48 h. Two prominent colonies were picked from the plate after incubation and grown in sterilized nutrient broth. Genomic identification of these microbial colonies was done by DNA sequencing at the Genomebio Lab at Pune, India.

Optimization of batch treatment time

The quantity of air and stirring speed were evaluated for optimization of PVA containing desizing textile wastewater degradation for minimum time. The selected acclimatized microflora of TSM was used as 5% inoculum in each experimental set contains one liter PVA containing actual textile wastewater. Air quantity varied from 8 to 32 l/min at 150 rpm and suitable air was optimized. The stirring speed was varied from 100 to 250 rpm at 16 l/min in separate experiments. As aeration was observed contributing maximum degradation, first optimization of air was done. After receiving the results of aeration optimization studies, stirring optimization studies were carried out for optimized air.

Continues scale studies of PVA containing textile waste degradation

Continuous study experiment set-up was prepared and ran the system on continuous mode by keeping 2 days HRT, 16 l/min air and 150 rpm stirrer agitation. PVA degradation and COD reduction were analyzed for a month in triplicates. Average readings of triplicates were used for analyzing results.

Analytical methods

COD measurement

The COD of water sample was measured according to the procedure described in Standard Methods (Clesceri *et al*. 1998). For rapid and efficient analysis of indirect COD by total carbon analysis of all PVA containing textile wastewater samples were done by TOC – V CPH (Shimadzu Ltd.) with a multiplication factor of 2.66 for final indirect COD quantification as explained by Magdum *et al*. 2013.

$$\text{Indirect COD (mg/l)} = (\text{TC or TOC reading mg/l} \times 2.66)$$

Iodine colorimetric method for PVA analysis (Finley 1961)

100-µl PVA containing the sample was taken and diluted to a volume of 10 ml. Add 5 ml of 4% boric acid and 2 ml of I_2-KI (1.27 g of I_2 and 25 g of KI in 1 liter). Solutions were equilibrated 5 min for each sample and then diluted to 25 ml with double distilled water. PVA concentrations were analyzed at 690 nm in a spectrophotometer (Model DR 2800, HACH, USA). All analyses were carried out in duplicates, and the average values were reported.

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Microbial Gram's staining and microscopy

Smears were prepared from respective microbial samples/swabs and these were stained using Jensen's modification of the Gram staining method (Duguid 1996). Stained slides were observed under oil immersion ($\times$100) magnification (Dewinter Trinocular Microscope – Model : Select).

RESULTS

Actual wastewater sample analysis

As shown in Table 1, colored effluent was highly alkaline; where as desizing effluent was acidic in nature. Both the effluents had the large difference in COD and BOD concentrations. Only 0.531% of PVA concentration was observed in the desizing effluent.

Table 1 | Analysis of colored and desizing effluent from actual textile industry

Parameters	Colored Effluent	Desizing Effluent
pH	11.89	3.76
COD (mg/l)	1,482.9	15,691.2
BOD (mg/l)	710	6,133.6
PVA %	NA	0.531
Chlorides (mg/l)	150.4	121.3
SO_4 (mg/l)	4.5	3.5
HCO_3 (mg/l)	42	100
Na (mg/l)	209.2	9

Isolation of selective microbial strains

Five microbial strains were grown on selective media AM1. By observing morphology, three bacteria with a yeast strain and a fungal species were found to be grown by utilizing (degradation) PVA as the only carbon source. These strains were further used for further selection studies with naming as TS1 (Bacterial strain), TS2 (Bacterial strain), TS3 (Yeast strain), TS4 (Fungal) and TSM (Mixed culture of TS1, TS2, TS3 and TS4).

Selection of effective PVA degraders

After successful isolation of PVA degrading microbial strains, further selection studies for the most effective PVA degrading strains for were carried out. After five days of constant shaking at 150 rpm and incubation at room temperature, good microbial growths were observed in all flasks (Figure 1).

After analysis of PVA degradation by Iodine method, all strains showed PVA degradation (Figure 2). The maximum PVA degradation was seen in TSM flask, which was 22.14% for 0.5% PVA, 28.57% for 1% PVA and 61.63% for 2% PVA in five days. PVA degradation efficiency can be arranged as TSM > TS2 > TS3 > TS4 > TS1.

Optimization of treatment process for maximum PVA biodegradation

Only aeration, only stirring and stirring with aeration, these three conditions were attempted to check the effect on PVA biodegradation rates. In these experiments, along with all isolated microbial strains,

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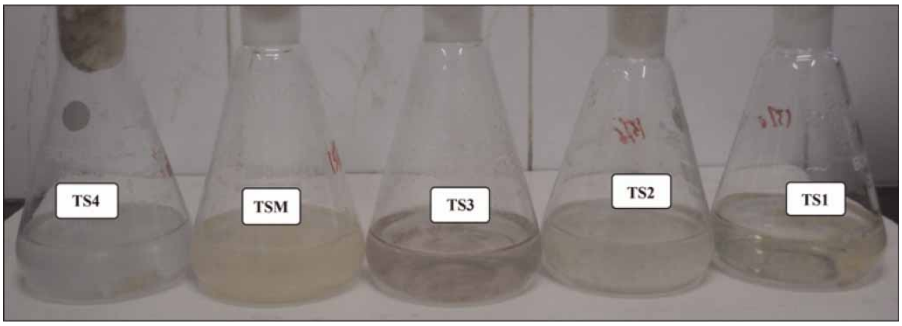

Figure 1 | Isolated microbial growth in a medium containing 0.5% PVA as a sole carbon source.

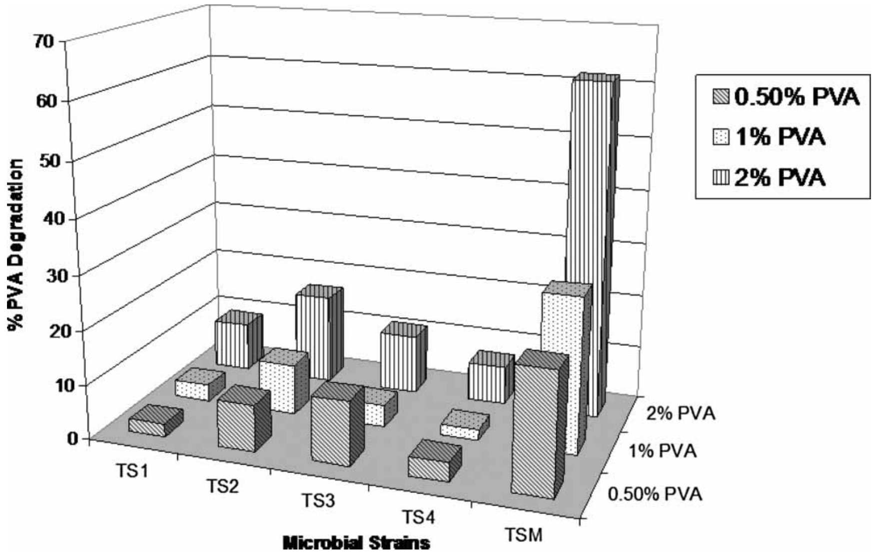

Figure 2 | Comparison of effective PVA degradation by isolated microbes TS1, TS2, TS3, TS4 and TSM (mixed culture of TS1, TS2, TS3 and TS4) with different PVA concentrations (0.5, 1 and 2%).

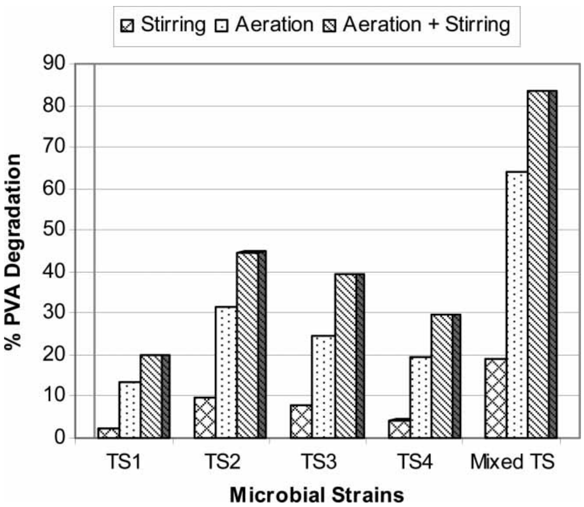

Figure 3 | Evaluation of three process treatment condition (stirring only, aeration only and stirring with aeration) for maximum PVA degradation.

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mixed strains were also evaluated for its degradation capabilities. All the five reactors of aeration with stirring showed the higher PVA degradation percentage (Figure 3).

PVA degradation in mixed TS (TSM) was 18.78, 64.2 and 83.55% for only aeration, only stirring and stirring with aeration conditions respectively, which were higher than all single strain inoculated reactors. TS2 showed higher, 44.8% PVA degradation in stirring with aeration than all other single strain inoculated reactors. Only aeration enhanced PVA degradation process by more than 3 to 4 times than stirring only. But simultaneous aeration and stirring further enhanced PVA degradation by 10 to 20% than with only aeration. Staining of microbial cultures showed (Figuer 4) that TS3 (Yeast strain) and TS2 (Bacterial strain) had dominantly grown in the TSM reactor while degrading PVA. In this experiment the mixed TS microbial consortia was worked symbiotically and found very efficient to treat PVA containing wastewater.

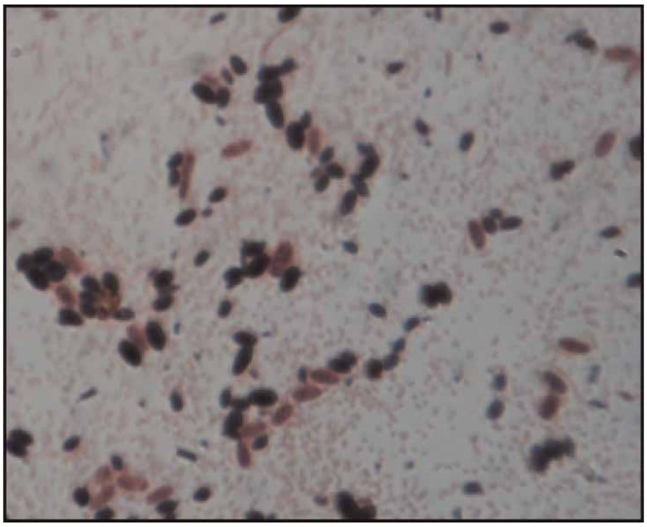

Figure 4 | Gram's staining of the PVA acclimatized culture of TSM.

Isolation and identification of acclimatized microbial strains

Two prominent microbial strains were re-isolated from TSM. Both the strains were identified based on ribosomal RNA sequencing. BLASTN 2.2.27 search of respective sequences were identified as *Candida Sp.* (Yeast) and *Pseudomonas Sp*. (Bacteria) with 97 and 98% maximum genomic identity respectively. This analysis acknowledged the microbial strains acting symbiotically for efficient PVA degradation. Various studies of PVA biodegradation had been done based on symbiotic microbial actions, such as, *Novosphingobium* sp. and *Xanthobacter flavus* (Duguid 1996), *Sphingomonas* sp. and *Rhodococcus erythropolis* (Vaclavkova *et al*. 2007). Experiments conducted by Nishikawa and Hasegawa resulted in identification of *Saccharomyces*, *Lipomyces* and *Rhodotorula* yeasts which degrade and assimilate PVA (Matsumura *et al*. 1999). *Pseudomonas* sp. has been known as PVA degrader, which produced PVA oxidase in symbiotic microbial treatment (Shimao *et al*. 1982).

Optimization of treatment time

For optimization of aeration and foam control, air was increased from 8 to 16 lpm/l at 150 rpm which was shown improvement in the initial PVA degradation rate by two times for day 1 and three times for day 2 (Figure 5).

At higher quantity of air (24 and 32 lpm/l), uncontrolled foaming (Figure 6) was observed. Therefore a high flow rate of aeration showed limitation causing immense polymer foaming. High aeration in PVA containing waste always creates the problem of foaming and leads to formation of PVA film to

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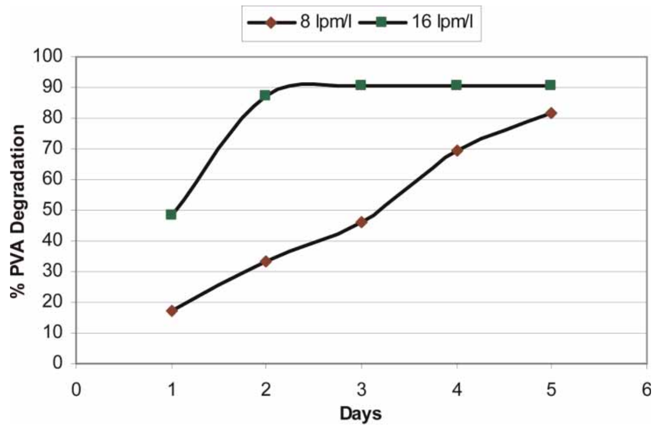

Figure 5 | Effect of different air flow rates (8 and 16 lpm/l) on % PVA degradation.

Figure 6 | Foaming during aeration optimization studies at 24 and 32 lpm/l aeration setups.

further clogging of carrier media used in treatment systems. Proteins present in the fermentation liquid from the outset, either as microbial product or in the form of extra-cellular polymers that are bound to solids (Ganidi *et al.* 2009).

Air optimization process reduced treatment time from 5 days to 2 days. Different stirring speed studies (at air 16 lpm/l) showed 150 rpm is the optimum speed for higher percentage PVA degradation, where 100 rpm showed lesser degradation. Stirring at 200 and 250 rpm did not add any prominent effect on PVA degradation (Figure 7). In case of the stirring rpm studies at with air 16 l/min and the stirring above 150 rpm did not give any significant difference in PVA degradation rates. Tsuchii & Tokiwa 2001 also observed an increase in the rate of degradation of rubber particles by increasing stirring speed.

Continuous biodegradation study

One month continuous PVA containing textile waste biodegradation studies showed consistent COD and PVA reduction on 2 days HRT (Figure 8).

Average inlet and outlet COD readings were 14,861 and 2227.65 mg/l respectively with 85.02% COD reduction. In case of PVA, average inlet and outlet percentage were 0.519 and 0.051% respectively with 90.3% of PVA degradation. It was reported that, when the concentration of

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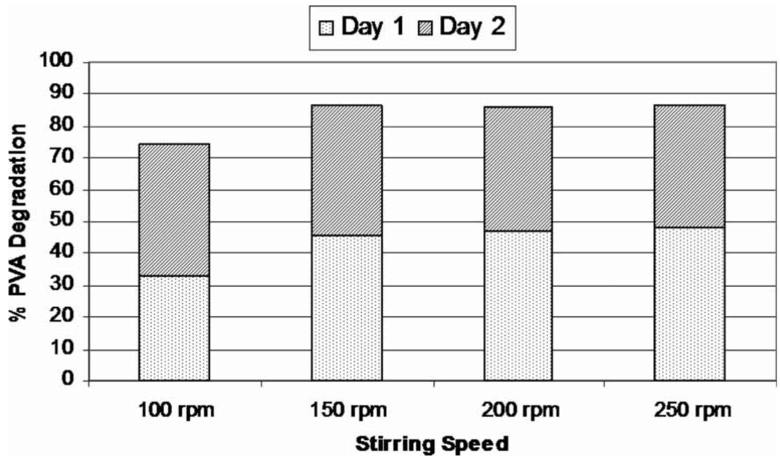

Figure 7 | Effect of different stirring speeds on % PVA degradation.

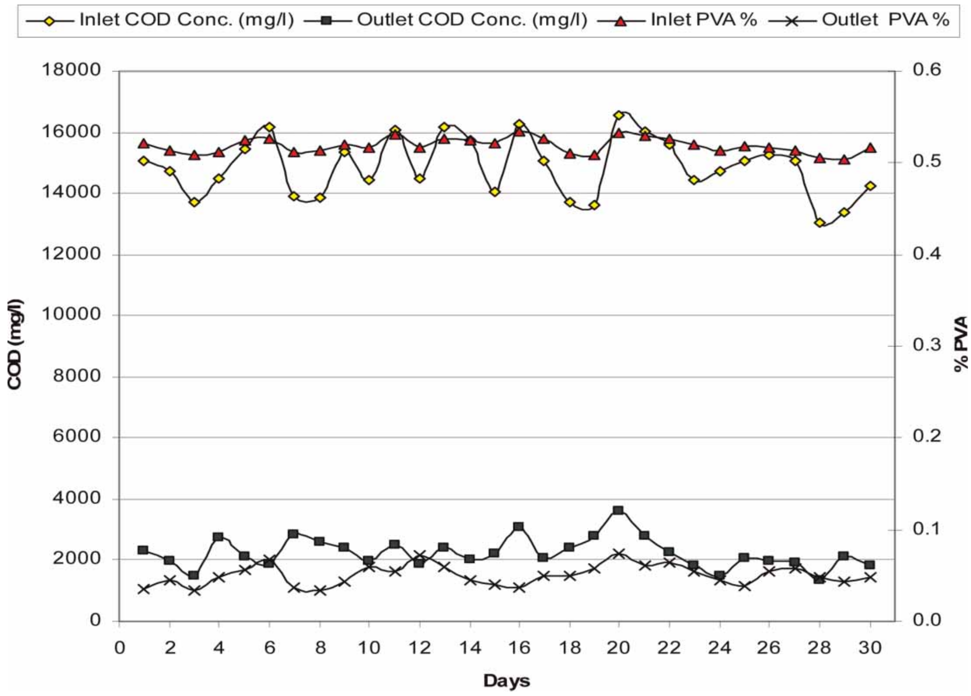

Figure 8 | Continuous biodegradation study of PVA containing actual textile wastewater.

PVA was 0.1, 0.5, 1 g/L respectively, more than 80% of PVA was degraded after 7 days and the removal of COD was 69.75% with proper treating time of 12 hrs (Chen *et al*. 2005). The study of Yamatsu *et al*. showed that, over 90% of PVA, at an initial concentration of 0.1%, was degraded within 6 days (Yamatsu *et al*. 2006). The comparison of viable treatment process of PVA containing textile waste, such as VFE (Gupta 2009), UF (Porter 1998; Altin 2008) and biodegradation have shown in shown in Table 2. UF and VFE need high initial investment and operating cost with other maintenance factors which are needed to be considered before installation. In the case of PVA recycle and reuse, the Indian textile industry has the preference for high quality textile processing than to reuse the PVA. Instead of high initial investment and textile quality risk of PVA reuse, it is always good to have better biodegrading treatment option as a worth standard process.

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Table 2 | Comparison of UF, VFE and biodegradation process for PVA containing wastewater treatment

	Biodegradation	UF	VFE
COD removal	95%	70–80%	80%
Water recovery	None	70%	70
Sludge problem	High	Low	Low
Maintenance and Energy Requirement	Low	High	High
Clogging problem	None	High	None
Advantages	• Low investment • Ease in operation • Reliable • Biological Process • Environment friendly	• Recycling of PVA and Water	• Recycling of PVA and Water • Water free from PVA
Disadvantages	• High HRT (2 days)	• High initial investments • High operating cost • Membrane maintenance • Stick to parameters • Lower efficiency • Specific recycling-lowering quality • Permeate contain PVA tails. • Depends on site location	• Very High initial investments • Very High operating cost • Dependant on site location

CONCLUSIONS

Two strains namely *Candida* Sp. and *Pseudomonas* Sp. were isolated microorganisms and they can be used to work symbiotically for efficient PVA degradation to treat PVA containing textile wastewater. Aerobic biodegradation of PVA containing wastewater can be achieved in 2 days of HRT with 90.3 and 85.02% removal of PVA and COD respectively with 16 lpm/l aeration and 150 rpm stirring. Process parameters such as aeration and stirring need to be optimized for different concentrations of PVA containing textile wastewater and for different organic loads. After comparing with other available treatment processes, present optimized process claims a simple (without any pre-treatment), highly efficient with least HRT, cost effective and environment friendly treatment solution for desizing wastewater.

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