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Dr. Sachin Madhavrao Kanawade
Dr.Vasantraodada Patil Shetkari Shikshan Mandal's Padmabhooshan Vasantraodada Patil Institute of Technology, Budhgaon, Tal – Miraj, Dist – Sangali, Maharashtra, India, Pin – 416304

Correspondence
Dr.Sachin Madhavrao Kanawade
Dr.Vasantraodada Patil Shetkari Shikshan Mandal's Padmabhooshan Vasantraodada Patil Institute of Technology, Budhgaon, Tal – Miraj, Dist – Sangali, Maharashtra, India, Pin – 416304

Pharmaceutical Effluent Treatment by Physicochemical Method

Dr. Sachin Madhavrao Kanawade

Abstract
A physicochemical study for the treatment of pharmaceutical wastewater was performed. Objective of the laboratory investigation was to study the removal of color, Total Dissolved Solids (TDS), Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), turbidity and phenol and bring them up to the allowable limits for reuse purposes. Efficiency of coagulation, flocculation, sedimentation, sand filtration followed by activated carbon adsorption was determined. It was found that tested coagulants (alum, ferric chloride, and ferrous sulphate) are not much effective and required high dosage for the removal; of TSS, BOD, COD and turbidity. Alum was found to be more effective among tested coagulants and reduce TSS, BOD, COD and turbidity 79.6%, 34.8, 48.6% and 69.2% respectively. Sand filtration further reduced the studied parameters 97.7%, 95.7%, 93.9% and 76.9% respectively. As the concentration of phenol in the studied pharmaceutical wastewater was 100 mg/l, granular activated carbon was used to remove phenol up to the allowable limit for reuse purpose. Activated carbon adsorption further reduces phenol, TDS, TSS, BOD, and COD up to 99.9%, 99.1%, 21.4%, 81.3% and 71.1% respectively. High removal of color observed after activated carbon adsorption. It was concluded that the suggested treatment scheme is suitable to bring the effluent quality up to the water quality standards.

Keywords: Pharmaceutical wastewater, physicochemical analysis, quality standards, reuses.

1. Introduction
Rapid industrialization, over the past 40 to 50 years, has resulted in the generation of increasing quantities of wastes and wastewaters containing high level of organic and inorganic pollutants. The growth of biological and pharmaceutical plants was greatly accelerated during and after World War II. Manufacturers of new products, particularly antibiotics, have greatly increased the waste treatment and disposal problems [Nemerow 1984] [10]. Pharmaceutical companies are one of the major contributors of hazardous and toxic effluents. Ireland, alone generates about 43 tons biological oxygen demand (BOD) from pharmaceutical industry [Henery *et al.* 1996] [6]. Similarly, in USA during 1983, total 35,52,000 tons of hazardous waste produced in which 200,000 tons of sludge produced by pharmaceutical industry only [Nemerow 1984] [10]. Wastes from pharmaceutical industry, producing penicillin and similar antibiotics are strong (high BOD and low pH) and generally cannot be treated with domestic wastewaters. Typical values of BOD, TSS and pH of wastewater produce during the production of antibiotic medicines are presented in Table 1. Pharmaceutical wastewater treatment often requires the information about the characteristic and composition of the waste

Table 1: Typical Characteristic of Antibiotic Waste [Nemerow 1984].

Characteristic	Waste from production of		
	Penicillin	Terramycin	General antibiotic
BOD, ppm	8,000-13,000	20,000	1500-1900
TSS	--	10	500-1000
pH	2-4	9.3	2-11

Treatment of Pharmaceutical Wastewater
Pharmaceutical wastewater if disposed with insufficient treatment may leads to great damage to the environment and groundwater resources. General treatment cannot be used for every pharmaceutical wastewater due to its variable composition. Therefore, specific treatment is required for specific type of wastewater.
Progress in wastewater treatment technology has been the result of laboratory and field scale experimentation largely on a trial and error basis [El-Gohary *et al.* 1995] [4]. A number to treatability study of wastewater from pharmaceutical industries have been done to treat the wastewater up to a sufficient degree.

Sand filtration, followed by chemical treatment is a proven procedure to treat the domestic wastewater for disposal as well as reuse [Ishaq and Al-Zahrani 2000] [7]. Studies suggested the similar methods for the treatment of pharmaceutical wastewater in addition to Granular Activated Carbon (GAC) adsorption. Based on survey conducted by US-EPA in 1998 [US-EPA 1992] [13], 10 of 244 responding pharmaceutical manufacturing facilities use GAC treatment to reduce concentrations of organic constituents (BOD and COD) in wastewater.

Since last several decades, local as well as foreign pharmaceutical industries are working in India. These industries are generating large volume of wastewater during production. Due to strict legislation and standards for wastewater treatment these industries are required to treat their waste accordingly. Characteristics of these pharmaceutical wastewaters differ widely. Therefore, a process selected for one type of wastewater may not be suitable for another. Keeping in view, a wastewater treatment study was performed for a typical pharmaceutical industry at Jeddah engaged in the production of anti-inflammatory drugs, Acecol-ACE inhibitor and several antibiotics.

2. Materials and Methods

Pharmaceutical wastewater was brought from the industry and laboratory scale study was carried out in the environmental engineering laboratory. Sand filtration set-up and other set-up accessories were fabricated in the central workshop at KFUPM. The present study was carried out to investigate the capability of physicochemical processes for the treatment of targeted pharmaceutical wastewater. Objective was to bring the effluent characteristics up to the reuse standards of US-EPA.

Table 2: Characteristics of pharmaceutical wastewater and typical domestic wastewater.

Parameter	Pharmaceutical Wastewater		Typical Domestic Wastewater*	
	Range of Values	Monthly Average Value	Range of Values	Monthly Average Value
pH	6.2 – 7.0	6.9	--	--
Color	--	Brownish	--	Gray to black
TDS	600 - 1300	1000	250 - 850	500
TSS	690 - 930	880	100 - 350	220
BOD	1300 - 1800	1600	110 - 400	220
COD	2500 - 3200	2800	250 - 1000	500
Alkalinity as $CaCO_3$	90 - 180	145	50 - 200	100
Turbidity	2.2 - 3.0	2.6	40 - 150	80
Phenol	95 - 125	100	--	--

* [Metcalf and Eddy 1991]

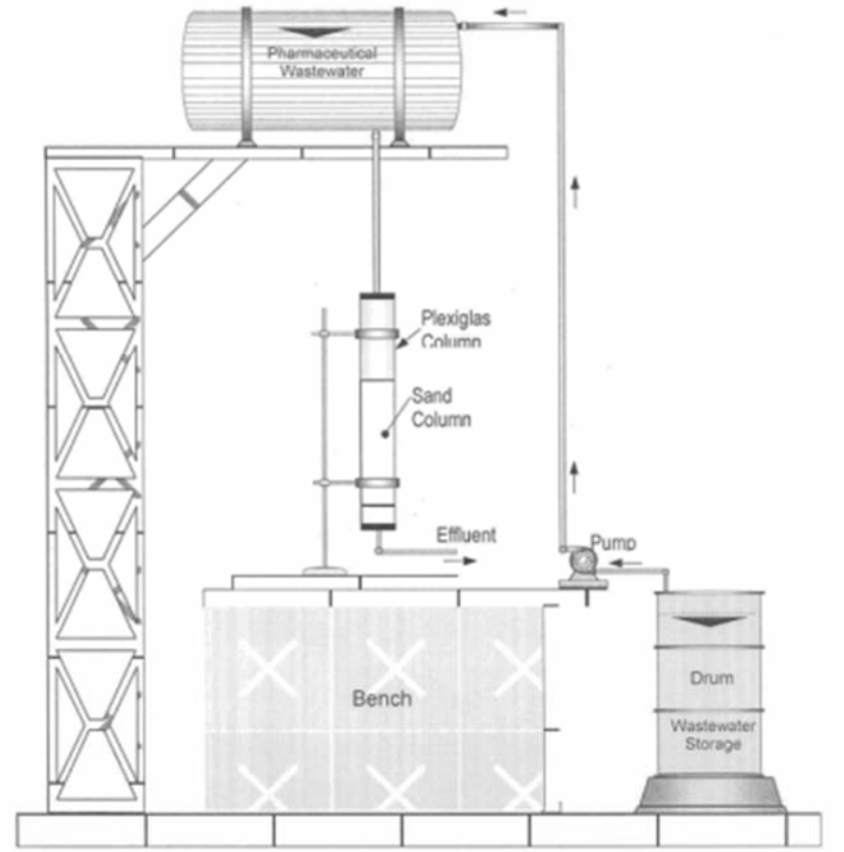

Fig. 1: Schematic diagram of the sand column setup (Down flow mode).

Characteristics of the pharmaceutical wastewater were determined (Table 2) and it was found that the strength of pharmaceutical wastewater is far more than the domestic wastewater in term of BOD, COD, TDS and phenol. A comparison of pharmaceutical and domestic wastewater characteristics is presented in Table 2.

Generally, pharmaceutical wastewater treatment may be done physically, chemically and/or biologically [US-EPA 1974]. In the present study physicochemical processes such as coagulation, flocculation, sedimentation, send filtration and activated carbon adsorption was evaluated. Common coagulants alum, ferric chloride, and ferrous sulphate were used for the preferable removal of SS, BOD, COD, and turbidity. In the case of ferrous sulphate and ferric chloride, the pH was maintained 9 to 9.5, while in the case of alum it was almost neutral. The optimum dose of coagulant was ascertained by conducting the jar test experiments. The supernatant from each Jar was analyzed for pH, color, TDS, TSS, BOD, COD, and turbidity. Sketch of the experimental unit used in the study is presented in Fig. 1.

Chemical treatment was followed by the sand filtration. Slow sand filtration was selected because it is well known process to improve the water quality. Slow sand filtration is preferred over the rapid sand filter because it is economical, practical, does not require skilled worker, and easy to maintain. A vertical circular plexiglass column of 5 cm diameter was filled with clean fine sand using the pluviation technique, creating a sand column of 30 cm height. Properties of sand used are presented in Table 3. The filtrate was analyzed for all the parameters studied in chemical treatment along with the phenol. As the filtration media used was fine sand, the limiting factor for the experiment was decided to be the clogging of filter.

Table 3: Physical properties of the sand used in the study.

Property	Value
Effective Grain Size (D_{10}) (mm)	0.16
Uniformity Coefficient (C_u)	1.85
USCS Classification	SP
Maximum Density, ρ_{max} (g cm^{-3})	1.82
Minimum Density, ρ_{min} (g cm^{-3})	1.54
Hydraulic Conductivity, K (cm sec^{-1})	0.012

Adsorption on activated carbon has been a useful and effective process for the purification of industrial and hazardous wastewaters, as well as for the removal of organic pollutants from water [Cyr *et al.* 2002]. High efficiency of the pollutant removal can be achieved because of large surface area available for pollutant adsorption. Large number of meso and micro-pores are produced during the manufacturing of activated carbon. These pore space is actually responsible for the formation of large adsorption surface area. Table 4 shows the surface area of several commercially available activated carbons.

Table 4: Surface areas of commercially available activated carbons

Commercial Activated Carbon Type	Surface Area ($m^2 g^{-1}$)
Filtrasorb 400	944
GRC-20	928
RO 3515	791
Hydrodarco GCW	874
Hydrodarco 4000	575

Pharmaceutical wastewater contains high concentration of organic and toxic pollutants, such as phenol, which may not be removed easily chemically as well as biologically. GAC is one of the most commonly used adsorbers for the treatment of phenol bearing wastewaters. Therefore, in the last phase of the treatment study GAC batch experiment was performed to polish the effluent quality and specifically get rid of high phenol concentration. Filtrasorb 400 GAC produced in USA, was used in the present study. An optimum dose of 1.0 g/l of GAC was used on the basis of previous study [Al-Zahrani *et al.* 2004] [2].

In addition to phenol removal, efficiency of activated carbon for TDS, BOD, COD and turbidity removal was evaluated. All analyses were conducted as per the Standard Methods for the Examination of Water and Wastewater [APHA *et al.* 1999]. In order to avoid temperature variation effects, all the experiments were performed at temperature between 21-22 °C.

3. Results and Discussion

Results of the present study consist of chemical treatment; sand filtration and GAC adsorption are discussed in the following section.

3.1 Phase I: Effect of Chemical Treatment

In the first phase jar test (coagulation, flocculation and sedimentation) was performed to find the effectiveness of studied chemical coagulants. A multiple stirring apparatus with variable speed drive was used for the study. In this physicochemical study, it was found that the effectiveness of studied coagulants is marginal except alum. The effect of dosage of three coagulants on the TSS removal is presented in Fig. 2. The optimum doses of ferrous sulfate, ferric chloride and alum for TSS removal of 79.6 to 67.7% was 240, 200 and 120 mg l^{-1} respectively. Turbidity removal even achieved at optimum dose of 10 to 20 mg l^{-1} for studied coagulants. A plot of turbidity variation with coagulant doses is presented in Fig.

3. It can be seen from Figs. 2 and 3 that at higher doses of coagulants the TSS and turbidity reduction was not significant. This could be due to the inorganic nature of suspended solids in the wastewater.

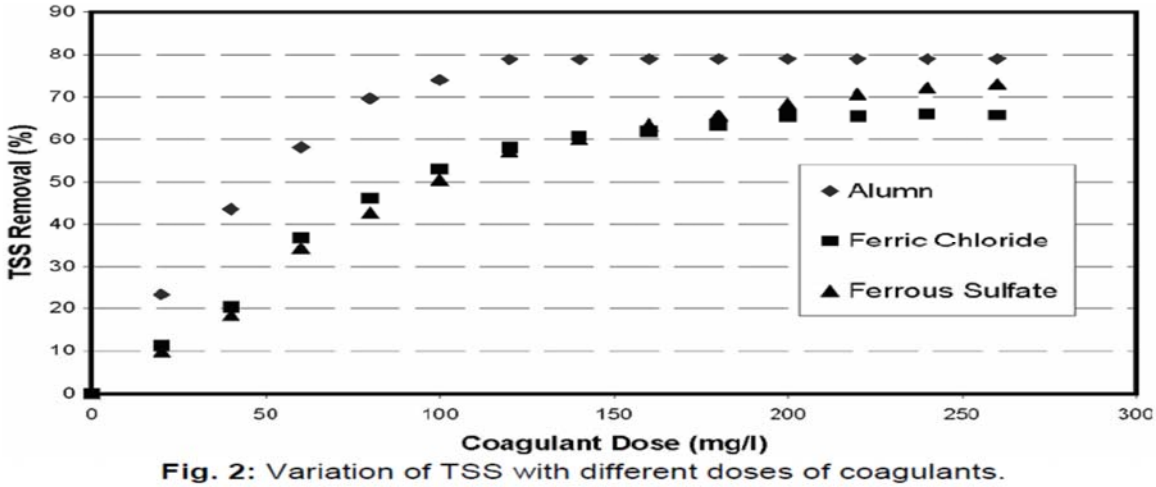

Fig. 2: Variation of TSS with different doses of coagulants.

Complete results of the analysis of effluent after chemical treatment (using determined optimum doses) are presented in Table 5. It is clear from the results that the alum is giving better quality effluent as compared to other two studied coagulants. However, the removal of BOD and COD is marginal (31.2 to 34.8% and 32.1 to 48.5% respectively).

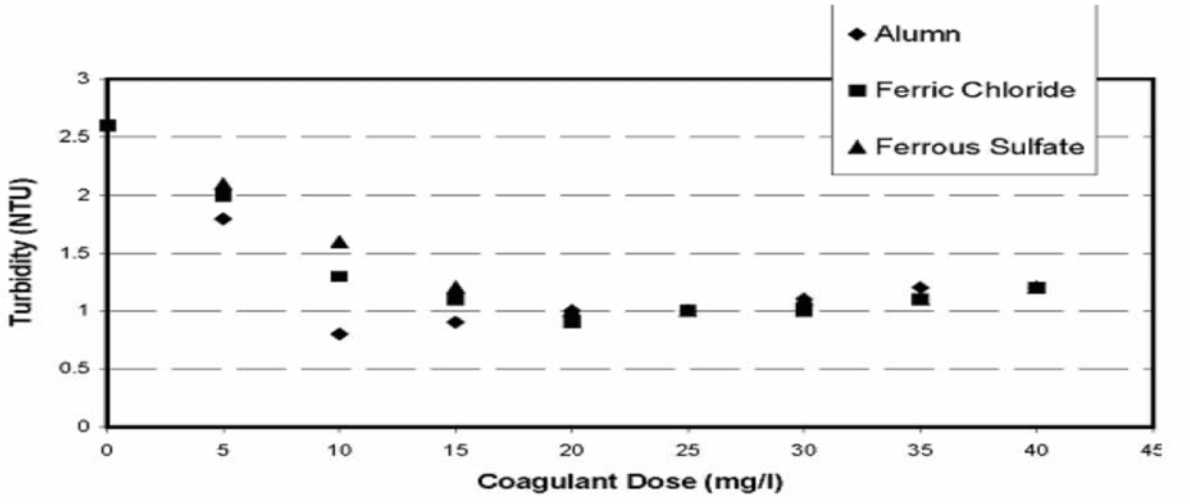

Fig. 3: Variation of Turbudity with different doses of coagulants.

Table 5: Comparison of effluent from different stages of treatment with the standards.

Para-meters	Raw Pharma-ceutical Wastewate	^Effluent After Chemical Treatment			*After Sand Filtration	After A.C. Adsorption	Saudi Arabian Standards			US-EPA [1992]
		1	2	3			MA [1989]	PME [2002]	RCJY [2003]	
pH	6.9	7.0	9.3	9.5	6.9	6.9	6.0-8.4	6.0-8.4	5-9	6-9
Color	Brownish	Light brown	Yellowish Brown	Gray-brown	Light brown	Color less	--	--	--	--
TDS	1000	580	600	605	548	4.8	--	--	--	--
TSS	880	185	289	205	4.2	3.3	20	15	15	5.0
BOD	1600	1043	1100	1080	45	8.4	20	25	50	10.0
COD	2800	1440	1560	1900	87	26	--	150	--	--
Turbidity	2.6	0.8	1.0	0.9	0.6	< 0.2	1.0	75	5.0	1.0
Phenol	100	-	--	--	73	< 0.002	0.002	0.1	--	

^ Wastewater treated with chemical coagulant, 1 = Alum, 2 = $FeCl_3$, 3 = $FeSO_4$,
* = effluent after chemical treatment with Alum used as influent. All units are in mg/l except color, pH, and turbidity.

3.2 Phase Ii: Effect of Sand Filtration

After chemical treatment the effluent was passed through the sand filtration unit with the help of an overhead tank arrangement from which influent was provided to the slow sand filtration unit by gravity. An overflow port was also provided in the column to divert the excess flow to a storage sump. Detail of the slow sand filtration column setup is presented in Fig. 1. The filter was operated by control of outlet flow. The filtration rate was kept to an average value of 0.15 m hr^{-1}. The rate of flow was controlled by an outlet valve, which had to be opened a bit further as the filter run progresses, to compensate for the increased hydraulic resistance of the filter bed.

The duration of the continuous experimental run was seven days. This short run was decided on the basis of findings of a three-year project on slow sand filtration as a tertiary treatment unit [Farooq and Nakhla 1997] [5]. Generally, the criteria for terminating slow sand filters runs is either the break through of turbidity or attainment of terminal head loss which subsequently results in a very dramatic decline of effluent flow rate. Therefore, the criterion for terminating filter runs was based on the clogging of the filtration unit irrespective of the quality of effluent.

Selected parameters for this study include variation of pH, turbidity, TSS, BOD, COD and phenol. Results show that pH did not vary significantly with time and it remained within the range of 7.0 to 6.9. Variation of studied parameters with time is depicted in Figs. 4, 5 and 6. After 168 hours of continues operation the value of turbidity was 0.6NTU. This low level of turbidity is an indication of good performance of the slow sand filter.

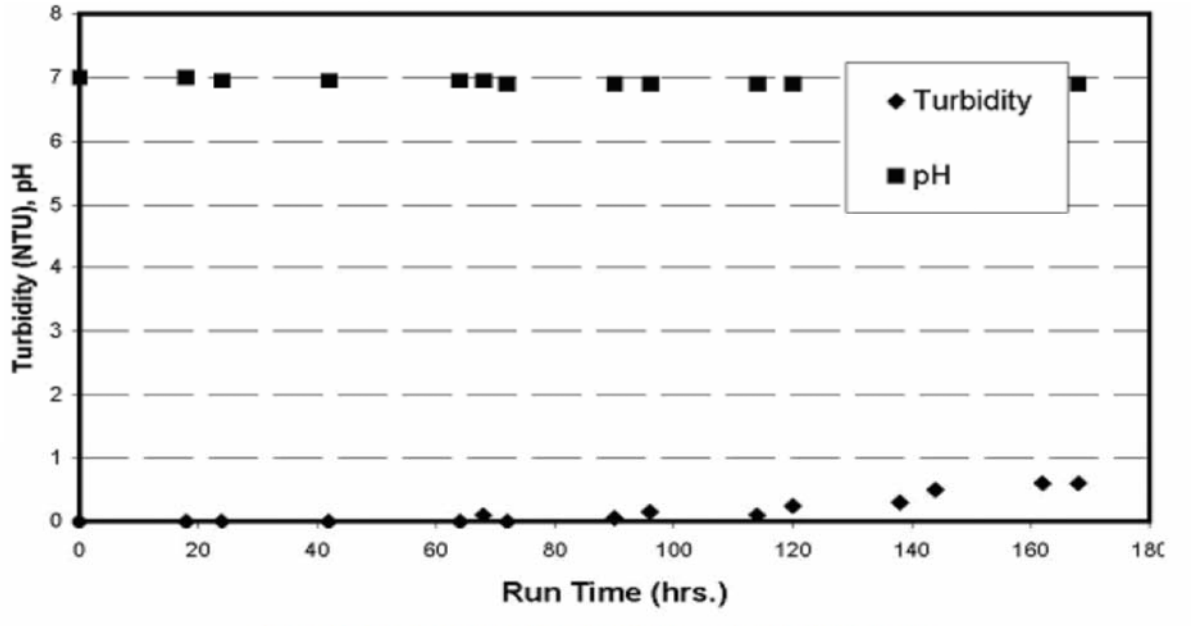

Fig. 4: Variation of Turbudity and pH with time.

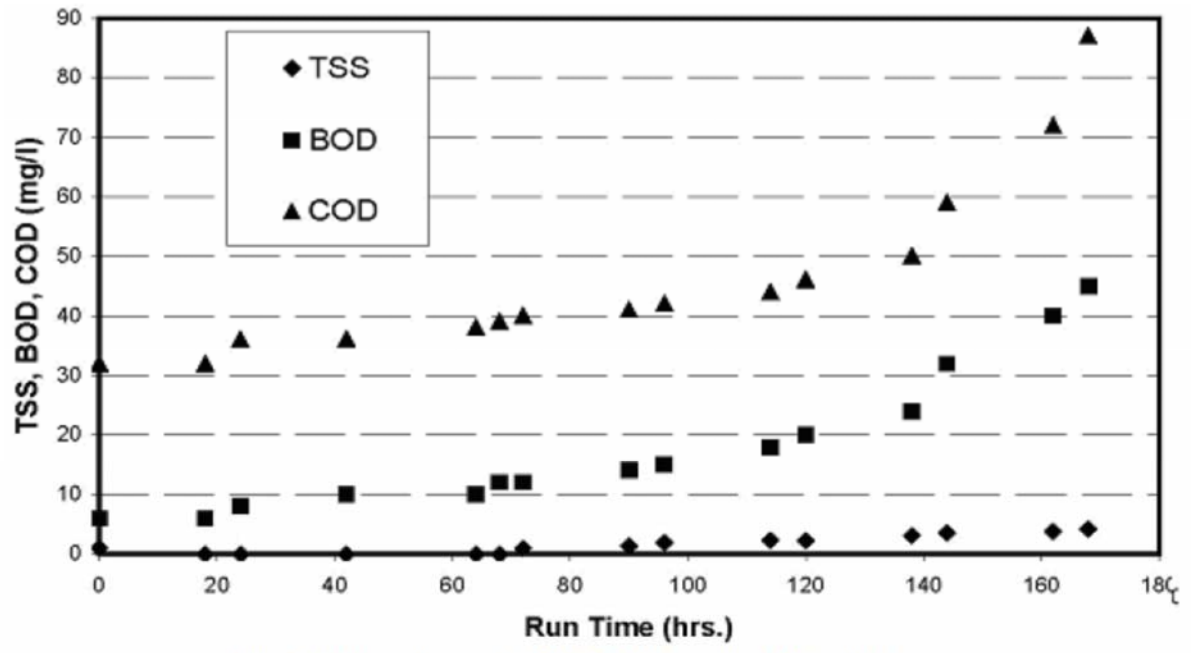

Fig. 5: Variation of TSS, BOD and COD with time.

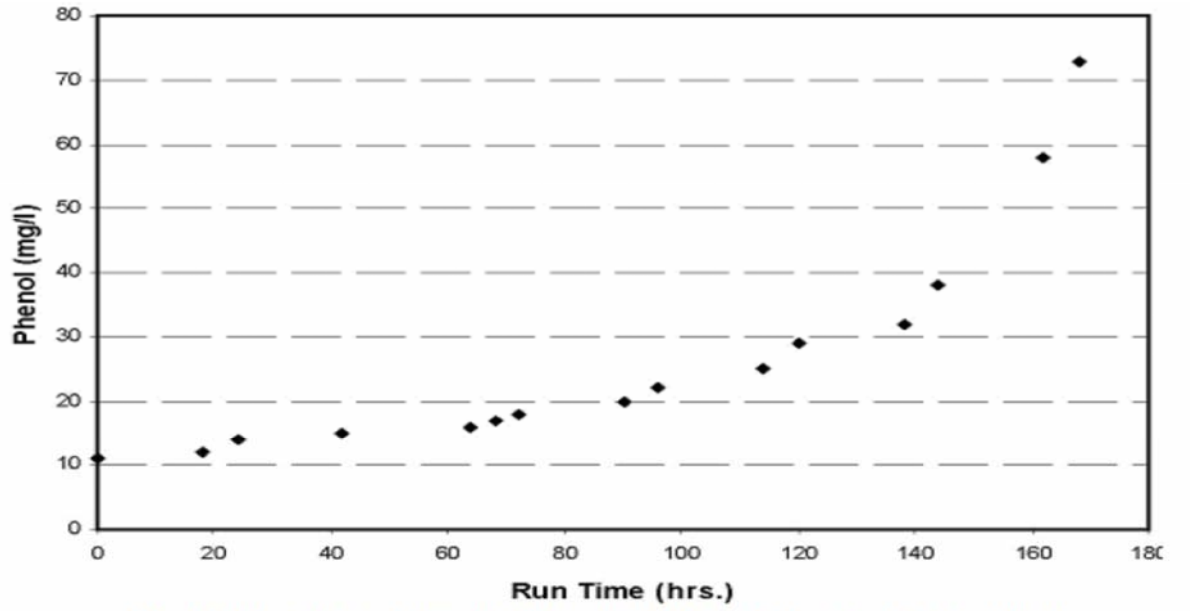

Fig. 6: Variation of Turbudity with different doses of coagulants.

High removal of TSS was observed during the run and at the end of the filtration run TSS value was 4.2 mg l^{-1} with a minimum removal efficiency of 97.7 %. Variation of TSS with time is presented in Fig. 5. Sand filtration system has long being recognized for organics removal. The influent water to the filter had BOD and COD concentrations of 1043 and 1440 mg l^{-1} respectively. It was found that the filter gave removal efficiencies for BOD and COD 95.7% and 94.0% respectively (Fig. 5). In the case of phenol removal, efficiency of sand filter was only 27% (Fig. 6). Summary of the results from chemical (coagulation, flocculation) and physical (sand filtration) treatment is presented in Table 5 along with the Saudi Arabian and US-EPA standards. Table shows that slow Sand filtration is a better mean of removing most of the organic and inorganic pollutants.

3.3 Phase Iii: Effect Gac Adsorption

GAC adsorption batch study was performed to bring the effluent quality up to the promulgated standard limits. Experimental runs were performed in the laboratory-controlled conditions. The pH of the influent and effluent was around 6.9 all the time. On the basis of studies elsewhere, contact time of 5 hours was kept for all batch runs [Al-Zahrani *et al.* 2004] [2].

It can be seen from the results presented in table 5 that GAC effectively reduced all the color, turbidity and phenol well below the limits and effluent is meeting the Saudi Arabian as well as US-EPA quality standards. Results also show that the GAC adsorption is important to use in order to remove the high phenol concentration in the pharmaceutical wastewater.

4. Conclusions

Proper treatment of pharmaceutical wastewater is important from public health and environmental protection point of view. Inherent diversity in each pharmaceutical wastewater forces the researchers to carry out a laboratory scale study in order to design and decide the treatment scheme. In the present study a combination of physical and chemical treatment was applied to a targeted pharmaceutical wastewater. Results suggest that alum could be use as chemical coagulant in the first stage of treatment scheme to remove TSS (79.6%), turbidity (69.2%) and a part of BOD (34.8%) and COD (48.6%). Sand filtration use after chemical treatment and gave high removal of TSS (97.7%), BOD (95.7%), COD (93.9), and turbidity (76.9%). GAC was used as the final stage in the treatment scheme. GAC adsorption brought the influent phenol concentration of 73 mg/l to less than 0.002 mg l^{-1} (99.9 %removal). In addition to BOD (94.2%), and COD (71.1%), high removal of color and TDS (99.1%) was observed. Study revealed that the combination of processes used in the studied is very well suited for the treatment of targeted pharmaceutical wastewater, which was highly concentrated and difficult to treat with other wastewaters. Treatment scheme is capable of bringing the effluent quality below the concentration limits set by US-EPA.

5. Acknowledgements

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Author's Biography:

Dr. Sachin Madhavrao Kanawade was born in 11 March 1978 at Nashik, Maharashtra, India. His native place is Nimgaonpaga, Tal-Sangamneer, Dist-A'Nagar, Maharashtra, India. He received his Bachelor's Degree in Chemical Engineering from Pravara Rural Education Society's Pravara Rural Engineering College, Pravaranagar (Loni) which is affiliated to Pune University in India in Nov.2001.Then he worked as a Production Officer in different Multinational Chemical Industries in India (2001 to 2008) like M/S Watson Pharma Ltd,Ambernath, MIDC, Mumbai, MS, M/S Glenmark Pharmaceuticals Ltd, Mohol, Dist. Solapur, MS, M/S Sun Pharmaceutical Industries Ltd, A. Nagar, MIDC,MS for 7 years.

Then he changes his field. He joined K. K. Wagh College, Nasik, MS, India in 2008 & worked as Lecturer for 2 years. At the same time he received his Master of Engineering in Environmental Engineering from Pravara Rural Education Society's Pravara Rural Engineering College, Loni in Dec.2010.Then he joined Pravara Rural Education Society's Sir Visvesvaraya Institute of Technology, Chincholi, Tal-Sinnar, Dist-Nasik, M.S. India in 2010 & worked as Assistant Professor in Chemical Engineering Department for 5 years. In the same period he completed his PhD Degree in Chemical Engineering in session 2011 – 2014 from Kumar Bhaskar Varma Sanskrit and Ancient Studies University Nalbari, Assam, India.

Presently he is Associate Professor at Dr. Vasantraodada Patil Shetkari Shikshan Mandal's Padmabhooshan Vasantraodada Patil Institute of Technology, Budhgaon, Tal – Miraj, Dist– Sangali, Maharashtra, India in Chemical Engineering Department.

Presently he is Reviewer / Editorial Board Member / Advisory Board Member of 64 different International Journals of different fields. He having 21 International Professional Membership of different Organizations

He published 47 Technical Research Papers in different International Journals like International Journal of Wastewater Treatment & Green Chemistry, International Journal of Chemical Engineering, International Journal of Environmental Pollution Control & Management, International Journal of Multidisciplinary Approach & Studies, International Journal of Chemical Engineering & Applications, International Journal of Chemistry & Material Science & International Journal of Engineering Studies and Technical Approach etc.

His research topic includes & interested in Chemical Engineering, Environmental Engineering, Wastewater Treatment by Adsorption, Advanced Separation Process, Chemical Engineering Design, Mass Transfer, Chemical Process Synthesis, Chemical Engineering Thermodynamics etc.