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CHARACTERISATION AND TREATMENT OF PHARMACEUTICAL R&D WASTEWATER

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ABSTRACT

A study was carried out on characterisation and treatment of wastewater discharged from a pharmaceutical R&D unit. The wastewater samples were collected from laboratory scale and pilot plant (scale-up) operations and analysed. Besides, combined wastewater samples were also collected and analysed. There is wide variation in waste characteristics due to the varied manufacturing operation/reactions employed. The combined waste exerts high BOD/COD value of 1385 mg/L and 5716 mg/L, respectively. The wastewater is treated in a full-fledged treatment plant comprising of equalisation, neutralisation, settling, extended aeration type biological treatment, pressure sand filtration and activated carbon filtration followed by a recycling plant with reverse osmosis and forced circulation mechanical evaporator. The wastewater samples were collected at various stages of treatment and results are presented.

INTRODUCTION

The Indian pharmaceutical industry today is in the front rank of India's science-based industries with wide ranging capabilities in the complex field of drug manufacture and technology. As per the draft national pharmaceutical policy 2006, Indian pharmaceutical market is estimated as Rs. 24,440 Crores (Shah, 2007). Since, Research and Development (R&D) forms the foundation of industry, the pharmaceutical companies invest a sizable sum in this area. The industry which now spends nearly three percent of its total sales on R&D activities is expected to increase it to five percent in the current fiscal year (Glenn Saldanha, 2007). Hitherto R&D was largely concentrated on process development for known drugs. After India signed the Trade Related Aspects of Intellectual Property Rights (TRIP) on 1995, the New Chemical Entity (NCE) research is also in the developing trend. To cater the research needs the corporate and multinational companies have their in-house and / or separate R&D facilities. The new innovator companies follow novel means to meet their R&D requirements, such as, collaborative approaches based on out-licensing and / or co-development of NCEs (Glenn Saldanha, 2007). As seen above, the pharmaceutical R&D is emerging as a fast growing industry.

The development of new drug requires the cooperative efforts of a large number of trained personnel specialising in medicinal, organic and analytical chemistry, microbiology, biochemistry, physiology, pharmacology, toxicology, chemical engineering and pathology. As a result of this diverse nature of pharmaceutical research and development, a wide range of chemical and biological laboratory wastes are pro-

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duced (US EPA, 1991). The diverse sets of waste streams make the waste complex in nature which poses problem of treatment.

The literature review on pharmaceutical waste characterization and treatment reveals the following reports. The physiochemical followed by biological treatment is suggested to treat pharmaceutical wastes by Alagarsamy *et al*. (1983), Ghosal and Bhowmik (1995), Das *et al*. (2000) and Shanta Satyanarayanan *et al*. (2004). The augmentation of existing pharmaceutical wastewater treatment facility with activated sludge system is reported by Deshmukh *et al*. (1984). A comprehensive wastewater treatment management for a basic drug industry is reported by Sateesh babu (1994). The anaerobic digestion followed by activated sludge process is reported to treat liquid waste arising from liver and beef extract manufacturing unit (Yeole *et al*. 1996). The physiochemical characteristics of drug industry waste and its influence on soil quality are reported by Bachewar and Mehta (2001). The unified solid flux theory is reported to improve the antibiotic wastewater treatment plant performance (Pophali *et al*. 2003). The electrochemical oxidation of pharmaceutical effluent is reported by Deshpande Abhijit *et al*. (2005). While reports on characteristics and treatment of pharmaceutical wastewater are extensively available, the report on pharmaceutical R&D waste is seldom available. Considering the same, the present study on characterisation and treatment of pharmaceutical R&D wastewater was undertaken.

MATERIALS AND METHODS

The present study was undertaken in a pharmaceutical R&D unit carrying out research activities on synthesis of new molecules / new chemical entity and identification of active ingredients in Indian plants and extracts them. The unit generates 10,000 litres (average quantity) wastewater per day.

The wastewater is treated in a full-fledged wastewater treatment and re-cycling plant. The wastewater is treated by physico chemical followed by biological treatment. The wastewater is equalised in a collection sump. The equalised waste is then pumped to the oil separation tank. After oil separation, the wastewater is drained out into neutralisation tank wherein acid or alkali is added to effect neutralisation. The neutralised waste is then allowed to settle in a primary settling tank. While the sludge settled is drained out into sludge drying bed, the supernatant is discharged into aeration tank at a controlled flow rate. The aerated mass is then taken to secondary settling tank. While the sludge / biomass settled is returned back to aeration tank, the overflow is collected in a sump. The treated waste is further polished by passing through pressure sand filter followed by activated carbon filter. The final treated wastewater is treated in re-cycling plant comprising of reverse osmosis system and mechanical evaporation system. The total dissolved solids and refractory organics in the treated wastewater are removed in the reverse osmosis system provided with plate-and-frame membrane modules. The permeate from reverse osmosis system is re-cycled and the reject is concentrated in a forced circulation evaporator. While the condensate from the evaporator is re-cycled, the concentrate is dried in a drier. The wastewater treatment and recycling plant provided at the unit is schematically shown in Figure 1 and 2.

The wastewater discharged from laboratory and pilot plant are separately collected. Besides, the combined wastewater, treated wastewater (reverse osmosis plant feed), reverse osmosis plant permeate and reject were separately collected. The samples collected were analysed as per the procedure given in Standard Methods for the Examination of Water and Wastewater (APHA, 1975).

RESULTS AND DISCUSSION

As stated earlier, the laboratory wastewater and pilot plant wastewater samples were separately collected and analysed. The analysis results of the same are presented in Table 1 and 2. The combined wastewater sample was collected from the collection cum equalisation sump of the wastewater treatment plant and the analysis results are shown in Table 3. The analysis results show wide variation in waste characteristics, attributed due to the varied operations and chemical reactions employed in R&D unit. The chemical synthesis, especially every step of an organic synthesis generates a mother liquor that contains unconverted reactants, reaction byproducts and residual product in the organic solvent base. An aqueous waste stream results from miscible solvents, filtrates, concentrates, equipment cleaning, wet scrubbers and spills (US EPA 1991). It is reported that the research related waste streams include inorganic acids and bases, organic solvents, metals, unused chemicals, reaction products from experiments and also waste oil from vacuum pumps and other rotating equipments (US EPA 1990). It is further reported

Table 1. Laboratory wastewater characteristics

Parameters	S_1	S_2	S_3	S_4	S_5	S_6	Avg.Value
pH	6.73	6.21	1.6	6.3	1.96	9.16	2.22
Suspended Solids	44	118	13	98	32	416	120
Total Dissolved Solids	696	1862	3024	8702	3640	3680	3601
BOD (3 days @ 27° C)	2352	2419	1176	1008	5.0	104	1177
COD	15237	10538	2856	7072	1994	8862	7760

All values except pH are expressed in mg/L ; S_1 to S_6 – Six samples collected at a time interval of 15 days.

Table 2. Pilot plant wastewater characteristics

Parameters	S_1	S_2	S_3	Avg.Value
pH	8.24	8.3	12.5	8.45
Suspended Solids	210	330	722	421
Total Dissolved Solids	5624	2452	257000	88359
BOD (3 days @ 27° C)	538	806	2083	1142
COD	14544	16864	193000	74803

All values except pH are expressed in mg/L; S_1 to S_3 – Three samples collected at a time interval of 15 days.

Table 3. Combined wastewater characteristics

Parameters	S_1	S_2	S_3	S_4	S_5	S_6	S_7	S_8	S_9	S_{10}	Avg.Value
pH	3.03	6.8	6.16	4.21	6.22	3.4	5.47	4.11	10.08	7.71	3.83
Suspended Solids	74	102	480	91	902	68	512	184	162	124	270
Total Dissolved Solids	5275	5050	3104	2356	4842	9328	7956	5100	3966	3468	5045
BOD (3 days @ 27° C)	660	2200	968	1024	2346	920	1025	1900	1932	880	1386
COD	4464	5472	5400	4096	8740	5800	4400	6624	5907	6256	5716

All values except pH are expressed in mg/L; S_1 to S_{10} – Ten samples collected at a time interval of 15 days.

Table 4. Treated wastewater characteristics

Parameters	S_1	S_2	S_3	S_4	S_5	S_6	S_7	Avg.Value
pH	7.48	7.29	7.03	7.63	6.79	7.14	7.15	7.14
Suspended Solids	176	36	188	58	64	28	206	108
Total Dissolved Solids	2606	3520	4948	1586	1834	6020	3012	3361
BOD (3 days @ 27° C)	154	26	123	83	193	72	123	111
COD	331	240	540	432	446	400	435	403

All values except pH are expressed in mg/L; S_1 to S_7 – Seven samples collected at a time interval of 15 days.

that the wastewater from synthesis processor typically have high BOD, COD and total suspended solids and pH from 1 to 11 (US EPA 1983). As expected, the pH of the combined wastewater varies from 3.03 to 10.08 due to residual acids and bases. Further, the combined waste exerts high BOD of 1386 mg/L and 5716 mg/L COD due to soluble and insoluble organics.

The analysis results of treated wastewater samples are furnished in Table 4. It is seen from the analysis results, the physico chemical followed by biological treatment effects partial reduction of BOD / COD. The residual BOD / COD in the treated wastewater is due to refractory / not easily bio-degradable organics. The analysis results of reverse osmosis permeate and reject samples are presented in Table 5 and Table 6 respectively. It is seen from Table 5, the suspended solids, total dissolved solids, BOD and COD in permeate samples are considerably brought down by reverse osmosis treatment. As expected, the reverse osmosis reject contains high total dissolved solids.

Table 5. Reverse osmosis permeate characteristics

Parameters	S_1	S_2	S_3	S_4	S_5	S_6	S_7	Avg.Value
pH	7.85	7.35	7.82	7.16	6.1	7.35	7.02	6.81
Suspended Solids	12	9.0	13	14	12	9.0	18	12.4
Total Dissolved Solids	312	240	340	252	90	170	230	233
BOD (3 days @ 27° C)	4.4	4.4	7.04	12.0	5.4	2.7	17.6	7.6
COD	25	56	72	154	46	20	95	67

All values except pH are expressed in mg/L; S_1 to S_7 – Seven samples collected at a time interval of 15 days.

Table 6. Reverse osmosis reject characteristics

Parameters	S_1	S_2	S_3	S_4	S_5	S_6	S_7	Avg.Value
pH	7.55	7.27	7.41	7.46	7.02	7.26	7.32	7.30
Suspended Solids	378	114	880	645	688	234	310	464
Total Dissolved Solids	8412	8976	17874	7316	4538	6646	5525	8470
BOD (3 days @ 27° C)	308	132	660	1564	506	117	308	514
COD	620	864	1980	3744	1380	700	952	1463

All values except pH are expressed in mg/L; S_1 to S_7 – Seven samples collected at a time interval of 15 days.

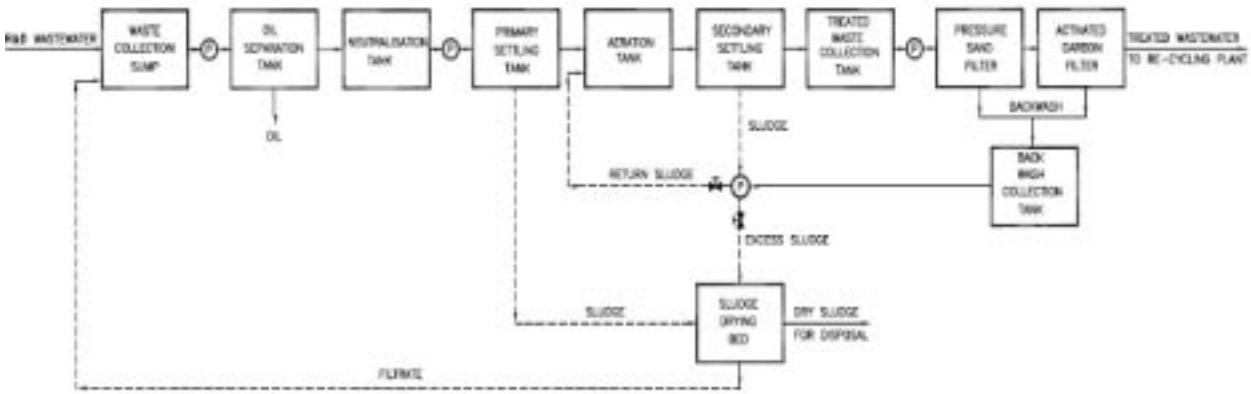

Fig. 1 Pharmaceutical R & D wastewater treatment scheme

Fig. 2 Wastewater re - cycling scheme

CONCLUSION

The study on characterisation and treatment of pharmaceutical R&D waste reveals that the wastewater contains high suspended and dissolved solids and also exerts high BOD / COD. The physico chemical followed by biological treatment effects partial reduction of BOD / COD. However, the reverse osmosis system removes the pollutants to a considerable extent and bring down total dissolved solids, BOD and COD and make fit the RO permeate for re-cycling.

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