

Treatment of Pharmaceutical Waste Water by Hybrid Separation Processes

E. Cséfalvay,^a K. Koczka,^a P. Mizsey,^a

^a*Department of Chemical and Environmental Process Engineering, Budapest University of Technology and Economics, HU-1111 Budapest, Hungary*

Abstract

Waste water from a pharmaceutical industry is treated by a hybrid separation process. The steps of the hybrid process are the following: i) conventional filtration to remove solid particles; ii) distillation to clean the waste water from volatile organic content; iii) nanofiltration and reverse osmosis to concentrate the not biodegradable components. The conventional filtration reduces the initial chemical oxygen demand (COD) of the waste water with ~14%, the distillation of solvents provides further ~45% reduction. The bottom product of distillation is treated by nanofiltration at first. Nanofiltration is proven to be not effective adequately because of the high COD of permeate and low flux. Reverse osmosis is applied then to concentrate the bottom product of distillation. Due to the high rejection of reverse osmosis, the not biodegradable organic compounds are enriched in the retentate of reverse osmosis, which energy content can be used in an incinerator plant. Permeate of reverse osmosis can either be lead to waste water pipe or further treated with biological tools.

Keywords: pharmaceutical waste water, distillation, hybrid separation process, membrane filtration

1. Introduction

Latest environmental regulations urge industrial companies to apply preventive environmental politics. In the pharmaceutical industry, however, the production of an active ingredient can not enable the technology to be changed basically. In this case, instead of preventive environmental politics, attention is focused on the treatment of the wastes. Since waste waters or process waters can not be minimized because of the long-standing technologies, effective end-of-pipe methods should be considered. [1] Usually, there are many engineering options for achieving the treatments. According to the EU Directives, biological treatment is aided. Nevertheless in certain cases the biological treatment can not be achieved but physical-chemical processes can offer a solution for this problem and they come increasingly to the front. [2, 3] Hybrid

processes including distillation combined with membrane techniques are promising alternatives for industrial waste water treatment, therefore more and more experiments are performed to support their industrial application. [4, 5]

The aim of the work is to give an optional process for the treatment of a pharmaceutical wastewater.

2. Experimental

2.1. Composition of waste water

The waste water treated is a process water of the production of an active ingredient. The ingredient is produced by fermentation therefore the waste water contains carbohydrates, a part of the feed of the fermentation, the product of fermentation and a slight concentration of solid particles. It contains among other compounds of high molecular weight, which are hardly biodegradable and it may contain traces of the ingredient. Since the ingredient production technology applies also solvent extraction, the waste water has a little solvent content, too. The waste water has a COD value of 42,000 mg/L.

2.2. Hybrid separation process

A schematic drawing of the applied treatment technology can be seen in Figure 2.1. The hybrid separation process consists of the following steps: i) conventional filtration to remove solid particles; ii) distillation to clean the waste water from volatile organic content; iii) nanofiltration and reverse osmosis to concentrate the not biodegradable components.

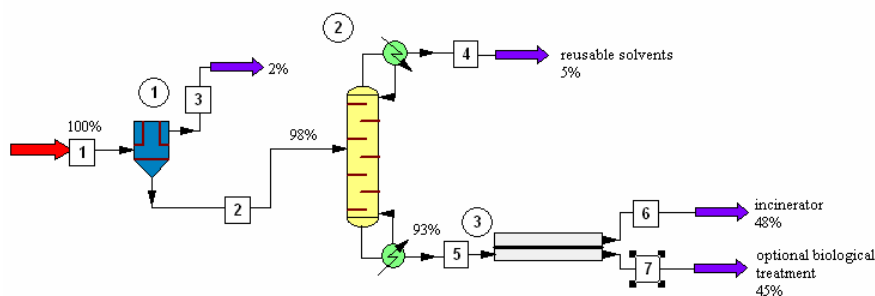


Figure 2.1: A schematic drawing of the applied treatment technology

2.2.1. Conventional filtration

In accomplish the removal of solvents, the solid particles need to be eliminated, since they can damage or disturb the subsequent part of the hybrid separation process. Filtration is carried out on a filter paper.

2.2.2. Distillation

After the conventional filtration the pre-treated waste water is fed into the bench-scale distillation column. The distillation column has 14 theoretical plates. The distillate/feed ratio is about 5%, the reflux ratio is 10. The distillate contains the volatile organic compounds, namely the solvents remained from solvent extraction and other organic compounds remained from the production of active ingredient. The high molecular compounds remain in the bottom product, which is to be further treated.

2.2.3. Membrane filtration

In the next step of the treatment, membrane filtration of the bottom product is carried out. Two different types of membranes are tested in order to determine whether the nanofiltration or the reverse osmosis membrane show better rejection. The applied membranes are: DK as nanofiltration membrane and SE as reverse osmosis membrane (made by Sterlitech Corporation). The COD of the permeate is determined and permeate flux as a typical parameter of membrane processes is also measured. The experiments are carried out on a bench-scale membrane apparatus with an effective membrane area of 28 cm².

3. Results and discussion

3.1. Conventional filtration

The waste water with a COD value of 42 000 mg/L is filtrated through a filter paper to remove the solid content of the waste water. This process enables the further treatment of waste water without damaging the system. Due to the removal of the solid particles the initial COD is reduced by 6000mg/L, i.e. 14.3%.

3.2. Distillation

Distillation is carried out to remove the volatile organic compounds (VOC) from the waste water. The distillate consists of two phases which ratio is 24:1, water rich phase: organic rich phase, respectively. According to the analysis by gas chromatography the organic rich phase contains solvents e.g. methanol, ethanol, isopropyl-acetate. The water rich phase contains traces of higher carbon-containing carbohydrates e.g. toluene. The COD measurements show that 45% of the initial COD can be removed by distillation.

3.3. Membrane filtration

The bottom product of distillation with a COD value of 17 000 mg/L is treated by membrane filtration. Nanofiltration is investigated at first. At room temperature and

10 bar only one-tenth of the optimal flux is observed. The membrane rejection is about 75%. In this case nanofiltration is proven to be not effective adequately.

Then a reverse osmosis membrane is also tested at room temperature and 30 bar. One-sixth of the optimal flux is observed. Both the nanofiltration and reverse osmosis membrane show much smaller flux than it is expected. The great decrease in flux can be explained by the high salt content of the waste water.

The reverse osmosis membrane shows a rejection of about 95%, which meets the typical rejection of reverse osmosis membranes. The COD value of the permeate increases during the experiment. Table 3.1 shows the COD values of permeate of reverse osmosis membrane in the yield of permeate.

Yield of permeate (%)	COD of RO permeate(mg/L)
20	973
40	2900
45	3070

Table 3.1: Chemical oxygen demand of the permeate of reverse osmosis membrane in the function of the yield of permeate

The permeate of the reverse osmosis membrane contains the not biodegradable compounds in negligible amounts therefore it can be further treated with biological tools. The Hungarian emission limit for the COD is 1000 mg/L so the first 20% of permeate can be lead into the waste water pipe. The energy content of the not biodegradable organic compound enriched in the retentate of reverse osmosis can be used in an incinerator plant.

4. Conclusions

The treatment of waste water streams containing low amount of volatile organic compounds is still quite difficult to handle. This study gives an alternative solution for the problem, presenting a hybrid separation process. The hybrid separation process consists of a conventional filtration, distillation and membrane filtration. Combining these processes, the generated hybrid process is proved to be efficient in waste water treatment. The quantity of the waste water can be minimized; the organic solvent content is removable and reusable, and biodegradable and not biodegradable components can be separated. The results satisfy the demand of latest EU Directives.

References

- [1] Mizsey, P., (1994) *Journal of Hazardous Materials*, 37, 1-13.
- [2] Getzer, M., (2002) *Journal of Cleaner Production*, 10, 305-319.
- [3] Belis-Bergouignan, M-C., Oltra, V. and Jean, M.S., (2004) *Ecological Economics* 48, 201-220
- [4] Koczka, K., Manczinger, J., Mizsey, P. and Fonyo Z., (2007), *Chemical Engineering and Processing* 46, 239-246
- [5] Roizard, D., Teplyakov, V., Favre E., Fefilatiev, L., Lagunstsov, N., Khotimsky, V., (2003), *Desalination* 162, 41-46