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Vincenzo Torretta[a], Maria Cristina Collivignarelli[b], Massimo Raboni[c] & Paolo Viotti[d]

[a] Department of Biotechnologies and Life Sciences, Insubria University of Varese, Via G.B. Vico, 46, I-21100 Varese, Italy

[b] Department of Civil Engineering and Architecture, University of Pavia, Via Ferrata 1, I-27100 Pavia, Italy

[c] School of Industrial Engineering, University LIUC-Cattaneo, Corso Matteotti 22, I-21053 Castellanza, VA, Italy

[d] Department of Civil and Environmental Engineering, University of Roma "La Sapienza", Via Eudossiana 18, I-00184 Rome, Italy

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Experimental treatment of a refinery waste air stream, for BTEX removal, by water scrubbing and biotrickling on a bed of *Mitilus edulis* shells

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[a]*Department of Biotechnologies and Life Sciences, Insubria University of Varese, Via G.B. Vico, 46, I-21100 Varese, Italy;* [b]*Department of Civil Engineering and Architecture, University of Pavia, Via Ferrata 1, I-27100 Pavia, Italy;* [c]*School of Industrial Engineering, University LIUC-Cattaneo, Corso Matteotti 22, I-21053 Castellanza, VA, Italy;* [d]*Department of Civil and Environmental Engineering, University of Roma "La Sapienza", Via Eudossiana 18, I-00184 Rome, Italy*

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The paper presents the results of a two-stage pilot plant for the removal of benzene, toluene, ethylbenzene and xylene (BTEX) from a waste air stream of a refinery wastewater treatment plant (WWTP). The pilot plant consisted of a water scrubber followed by a biotrickling filter (BTF). The exhausted air was drawn from the main works of the WWTP in order to prevent the free migration to the atmosphere of these volatile hazardous contaminants. Concentrations were detected at average values of 12.4 mg Nm^{-3} for benzene, 11.1 mg Nm^{-3} for toluene, 2.7 mg Nm^{-3} for ethylbenzene and 9.5 mg Nm^{-3} for xylene, with considerable fluctuation mainly for benzene and toluene (peak concentrations of 56.8 and 55.0 mg Nm^{-3}, respectively). The two treatment stages proved to play an effective complementary task: the water scrubber demonstrated the ability to remove the concentration peaks, whereas the BTF was effective as a polishing stage. The overall average removal efficiency achieved was 94.8% while the scrubber and BTF elimination capacity were 37.8 and 15.6 g BTEX d^{-1} m^{-3}, respectively. This result has led to outlet average concentrations of 1.02, 0.25, 0.32 and 0.26 mg Nm^{-3} for benzene, toluene, ethylbenzene and xylene, respectively. The paper also compares these final concentrations with toxic and odour threshold concentrations.

Keywords: air pollution; benzene; biotrickling filter; scrubber; toluene; VOCs

Introduction

Benzene, toluene, ethylbenzene and xylene (BTEX) are typically contained in petroleum and its derivatives, mainly gasoline and diesel. Therefore, they are found in significant concentrations in refinery as well as chemical and petrochemical industries wastewaters. Furthermore, BTEX are used in many other industries, such as pharmaceutical, plastics, dyes, resin-glues and cosmetics. Because of their high volatility, BTEX are also present in waste air streams generated by these industries and by all activities that use these compounds as paint thinner or degreaser. BTEX are also found in the biogas from municipal and industrial waste landfills [1–3] and in urban air,[4–6] mainly because of the vehicles' exhaust gases.[7–11] BTEX are also present in the exhaust air of many water and wastewater stripping processes.[12–14]

Due to their high toxic and genotoxic properties,[15] it is essential to minimize their migration into the environment and, above all, into the atmosphere, in order to protect workers and population health.

Waste air streams containing BTEX are normally treated using several physical–chemical and thermal processes, such as adsorption, condensation, incineration, as well as thermo-catalytic and chemical oxidation.[16–18] These processes achieve high removal efficiencies. The drawbacks are the high investment and operating costs. Actually, water scrubbing is a viable low-cost option, but it is effective only as a rough pre-treatment of medium- to high-polluted air streams.

Recently, biological processes such as biotrickling and biofiltration gained increased attention due to their low cost and energy efficiency [19]; moreover, they proved to be very effective in treating several industrial air streams. The most known applications are in the field of odour removal from exhaust air of wastewater treatment plants (WWTPs), composting and solid waste treatment plants.[20–24] The importance of these biological processes has been recently emphasized because of their ability to treat air streams with hardly degradable contaminants at low concentration.

Biotrickling filters (BTFs) show several advantages over biofiltration technologies for air pollution control: lower bed height limitation; smaller footprints; packing longevity over 10 years; lower pressure drops due to high media porosity; easy control of temperature, pH, salts concentration and metabolites accumulation and wider range

*Corresponding author. Email: vincenzo.torretta@uninsubria.it

of treatable pollutants.[19] For all these features, BTFs are used alone or in combination with conventional biofilters.

The aim of this paper is to demonstrate the effectiveness of a two-stage pilot plant for the treatment of refinery WWTP off gases, characterized by peaks and strong fluctuation in the BTEX concentration. The water scrubber as a first stage has been chosen for the removal of peak concentrations, whereas the BTF as a second stage has been chosen for its polishing role, which is very important for the reduction of BTEX under toxic and odour threshold concentrations.

Materials and methods

Pilot plant description

The pilot plant is composed of two treatment stages (Figure 1):

- a water scrubber (packing volume: 4.0 m³; packing height: 1.6 m; diameter: 1.6 m) filled with 2 in. Pall Rings;
- a BTF (bed volume: 6 m³; bed height: 1.0 m) filled with waste blue mussel (*Mitilus edulis*) shells. Such packing material exhibits a high buffering power due to their chemical composition ($CaCO_3$), useful to prevent an excessive lowering of pH in the biological process, which could inhibit the bacterial activity.

The pilot plant was fed with 600 $Nm^3\,h^{-1}$ waste air stream drawn from the main works of a refinery WWTP (mainly pumping station and primary settling tanks). Such a flow rate has been chosen in order to achieve a 24-s contact time into the scrubber, useful for an effective removal of the inlet peak concentrations.

The WWTP effluent (based on a biologically activated sludge process) was used as washing water and water make-up for the scrubber and the BTF, respectively. This choice has been made with a dual purpose: (i) supplying the BTF bacteria with the required nutrients and (ii) favouring the biological activity through the inoculation of bacteria acclimated to the specific substrate. Scrubber washing water and BTF trickling liquid flow rate were set at 3.0 and 0.9 $m^3\,h^{-1}$, respectively. Trickling liquid was continuously recirculated (hydraulic residence time: 7.5 d) in order to ensure a high wetting degree and to promote the oxidative biological degradation. During the plant start-up, a selected consortium of microbial population was inoculated into the BTF in order to speed up the full operation of the biological process. The inoculum was prepared by feeding for 20 days a small laboratory-activated sludge plant (100 $L\,h^{-1}$ capacity) with the effluent of the refinery WWTP and dosing BTEX, so as to achieve gradually increasing concentrations. Activated sludge initially collected from the refinery WWTP had the following characteristics: volatile suspended solids = 2.4 $kg\,m^{-3}$; dissolved oxygen = 1.85 $mgO_2\,L^{-1}$; sludge volume index = 188 $mL\,gSS^{-1}$; oxygen uptake rate = 77.5 $mgO_2\,L^{-1}\,h^{-1}$. After this period, 5 L of the activated sludge was added to the BTF water sump.

Research mainlines and analytical methods

The pilot plant ran for a period of 100 days, during which a sampling and analysis campaign was carried out. The campaign was conducted after setting the plant in regular operation (mainly to allow the growth of an acclimated bacteria consortium over the BTF bed). During the regular operation, 35 samples were taken upstream and downstream at each stage (total number of samples: 105).

In a second phase of the experimentation, the air flow rate changed in the range 300–600 $Nm^3\,h^{-1}$ in order to evaluate the effect of the empty bed retention time (EBRT) and, as a consequence, the air/water ratio on the scrubber performance.

The air samples were collected onto suitable sorbent tubes (ORBO 32) using SKC sample pumps.

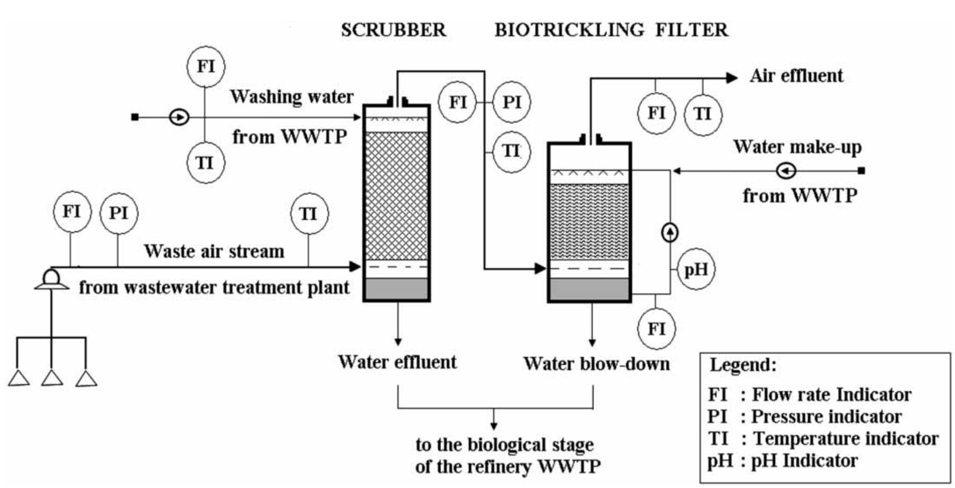


Figure 1. Diagram of the two-stage pilot plant.

Following desorption, the samples were analysed by gas chromatography-mass spectrometry (Varian Gas Chromatograph equipped with a high-resolution capillary column interfaced with a Finnigan Matt ITS40 Mass) in order to determine BTEXs (limit of detection: 0.05 mg Nm^{-3}). Temperature (T) and flow rate (Q) were measured using a Delta Ohm HD 2303.0 Hot Wire Anemometer with an AP471 S1 probe (accuracy: ±0.1°C for T; 0.5% of full scale for Q).

Sampling and analysis on the liquid effluent of the scrubber were performed in compliance with the standard methods.[25]

The identification of the consortium of bacteria responsible for biodegradation in the BTF was carried out. The community DNA was extracted from samples collected from the BTF and used as the template for 16S rDNA amplification with bacteria as universal primers.[26] Amplicons were separated by denaturing gradient gel electrophoresis. The more abundant ones were recovered from the gel and sequenced. The detected sequences were then compared with those present in GenBank.[27]

Results and discussion

Quality of the waste air stream and objective of the depuration

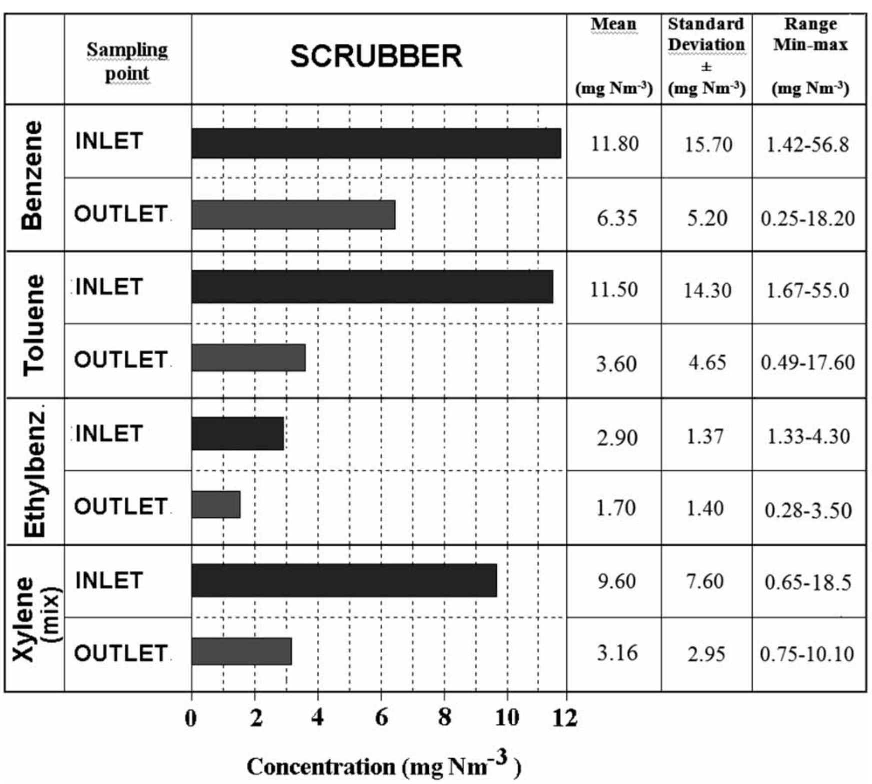
The quality of the waste air stream (as mean, standard deviation and range of detected data) is shown in Figure 2. A very wide variability of the concentrations can be observed around the average values of all compounds, especially benzene and toluene. The two compounds were measured at peak concentrations above 50 mg Nm^{-3}, which are five times higher than their average values. Total xylene was found at the average concentration of 9.60 mg Nm^{-3}, while much smaller was the average concentration of ethylbenzene (2.90 mg Nm^{-3}). The inflow air temperature was in the range 23–27°C (mean: 25.0°C and standard deviation: 1.6°C).

BTEX have low solubility in water. However, benzene has a value (1780 mg L^{-1} at 25°C) well above that of other compounds.[15,28] The values of vapour pressure indicate their high volatility, which is particularly pronounced for benzene and toluene (9999 and 3786 Pa at 25°C, respectively). This is the reason why they are present, at the concentrations found, in the waste air stream.

Table 1 shows the air exposure threshold concentrations for BTEX. They represent the technical target of the experimented treatment to preserve human health from toxicological risks or more simply odorous nuisances.

The threshold concentrations indicate benzene as the most dangerous to human health; in particular, stand out the very low TWA value of 1.7 mg Nm^{-3}. Table 1 also reports the olfactory threshold concentrations which are significantly small for xylene (mix) and toluene.

A comparison between the concentrations found in the waste air stream and the toxicological limits highlights the



	Sampling point	Mean (mg Nm^{-3})	Standard Deviation ± (mg Nm^{-3})	Range Min-max (mg Nm^{-3})
Benzene	INLET	11.80	15.70	1.42-56.8
	OUTLET	6.35	5.20	0.25-18.20
Toluene	INLET	11.50	14.30	1.67-55.0
	OUTLET	3.60	4.65	0.49-17.60
Ethylbenz.	INLET	2.90	1.37	1.33-4.30
	OUTLET	1.70	1.40	0.28-3.50
Xylene (mix)	INLET	9.60	7.60	0.65-18.5
	OUTLET	3.16	2.95	0.75-10.10

Figure 2. Scrubber performance for single BTEX removal. Bars represent the average value, whereas the table reports: mean (m); standard deviation (sd); range (min–max).

Table 1. Safety and od our exposure threshold concentrations for BTEX.

Substance	IDLH[a] ($mg\,Nm^{-3}$) [28]	STEL[b] ($mg\,Nm^{-3}$) [29]	TWA[c] ($mg\,Nm^{-3}$) [29]	Odour[d] ($mg\,Nm^{-3}$) [28]
Benzene	1'742	8.7	1.7	14–24
Toluene	2'055	411.1	205.5	1
Ethylbenzene	3'789	592.0	473.6	663
Xylene (mix)	4'263	710.5	473.7	0.2

[a]IDLH: Immediately dangerous to life and health (maximum concentration from which a worker should go away within 30 min before experiencing symptoms able to prevent their escape or irreversible effects on health).
[b]STEL: Short-term exposure limit (weighted average exposure over a period of 15 min which should never be exceeded in the workday).
[c]TWA: Time weighted average (weighted average concentration over time on a working day of 8 h and 40 h $week^{-1}$, to which nearly all workers may be repeatedly exposed, day after day, without adverse effects).
[d]Odor: Olfactory threshold concentration (concentration at which the odour is perceived by a panel of sniffers).

need for a drastic removal of benzene and, to a lesser extent, of the other compounds. Regarding toluene and xylene, a strong removal is also necessary to avoid the perception of odorous nuisances (toluene: aromatic, like benzene; xylene: sweet).

Water scrubber performance

Figure 2 shows the performance of the scrubber in removing BTEX.

The scrubber achieves a fairly good removal of BTEX, because of the higher contact time with respect to the traditional scrubbers and, above all, the high level of solubility in water (Figure 3).

The most striking aspect concerns the higher removal efficiency of toluene and xylene compared with benzene, if we consider that the latter has a much higher solubility in water. This result is only seemingly anomalous, since during the experimentation, a noticeable bacterial growth on the packing was found, hence requiring an intensive washing to remove the excess biofilm (the pressure drop due to clogging reached the maximum value of 60 mmH_2O). In fact, the toluene has characteristics of better biodegradability compared with benzene and ethylbenzene as widely documented in the literature. In particular, it has been reported that the same bacteria are able to degrade toluene and benzene, but when the two compounds are present together, they exerted an antagonist effect, so that benzene is degraded at a lower rate than toluene [30–32]; in particular, it was demonstrated that *Pseudomonas putida* in the presence of a mixed substrate is able to degrade toluene at a lower rate than that of toluene alone, due to the inhibitory effect exerted by benzene. Furthermore, a report edited for US-EPA concluded that benzene has a biodegradation constant rate at 20°C of 0.096 d^{-1}, while toluene proved a much higher value (0.2 d^{-1}); for ethylbenzene and xylene the constant rates were found equal to 0.113 and 0.055 d^{-1}, respectively.[33]

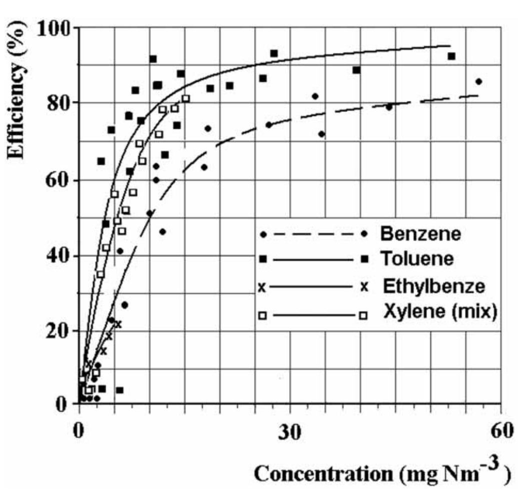


Figure 3. BTEX removal efficiency in the scrubber unit as a function of the inlet concentration. Curves represent the single BTEX average removal efficiency.

Due to the strong fluctuations of the inlet concentrations, the BTEX average removal efficiency resulted in a very wide range: 46.2% for benzene; 68.7% for toluene; 41.4% for ethylbenzene and 67.1% for total xylene. The efficiency referred to total BTEX has amounted to 58.6%: a mass balance evidenced that about one-fourth (15.1%) is due to absorption, whereas the remaining part (43.5%) is due to the biological degradation.

Figure 4 shows the results of the second phase of the experimentation, which was carried out for evaluating the influence of different air/water ratios on the scrubber performance.

The results show the benefit of low air/water ratio on the removal efficiency of total BTEX. In fact, the lower the air/water ratio, the higher the EBRT of the contaminants and the efficiency of washing. High removal efficiencies (greater than 70%) are achieved with air/water ratio lower than 160 $Nm^3\,m^{-3}$ (EBRT $>$ 30 s).

BTF performance

Figure 5 shows the performance of the BTF.

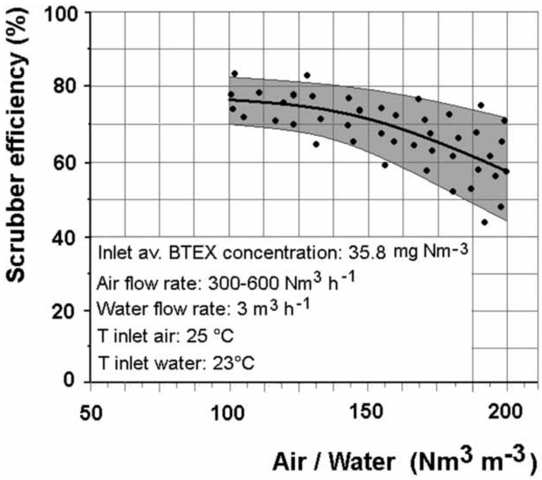


Figure 4. BTEX removal efficiency of the scrubber as a function of the air/liquid ratio. The continuous line and the shaded area represent the mean and the 95% confidence interval, respectively.

The passage through this stage has determined the further reduction in the BTEX concentrations, allowing one to achieve average removal efficiencies of 83.9% for benzene, 93.1% for toluene, 81.2% for ethylbenzene and 91.8% for total xylene. The average efficiency, referred to total BTEX, has amounted to 87.5%.

In the consortium of bacteria responsible for biodegradation, several species of the genus *Pseudomonas*, *Ralstonia*, *Dechloromonas* and *Acinetobacter* were identified.

During the experimentation period, the temperature of the water recirculated over the filter was in the range 22–25°C and the pH in the range 7.4–7.6. This latter finding is noteworthy because it has been made possible by the buffering action of the blue mussel shells rich in limestone, whose reaction with carbon dioxide, produced by the biodegradation, has prevented the lowering of pH in an acidic field, with a consequent inhibition of the bacterial activity. This phenomenon has determined the need of periodical make-up of the shells.

Figure 6 shows the biotrickling removal efficiency as a function of the volumetric load referred to the total BTEX.

The graph proves that volumetric loads lower than 30 g BTEX d^{-1} m^{-3} allow one to achieve removal efficiencies higher than 90%.

Figure 7 shows the elimination capacity of the BTF as a function of the volumetric load.

Due to the very low inlet concentrations of BTEX, the initial part of the curve has a rectilinear course, while at about 35 g BTEX d^{-1} m^{-3} the curve tends to have a progressively smaller gradient. However, in correspondence with volumetric loads of 10–60 g BTEX d^{-1} m^{-3}, the specific removal capacity has resulted in the range 10–46 g BTEX d^{-1} m^{-3}. It should be noted that these figures

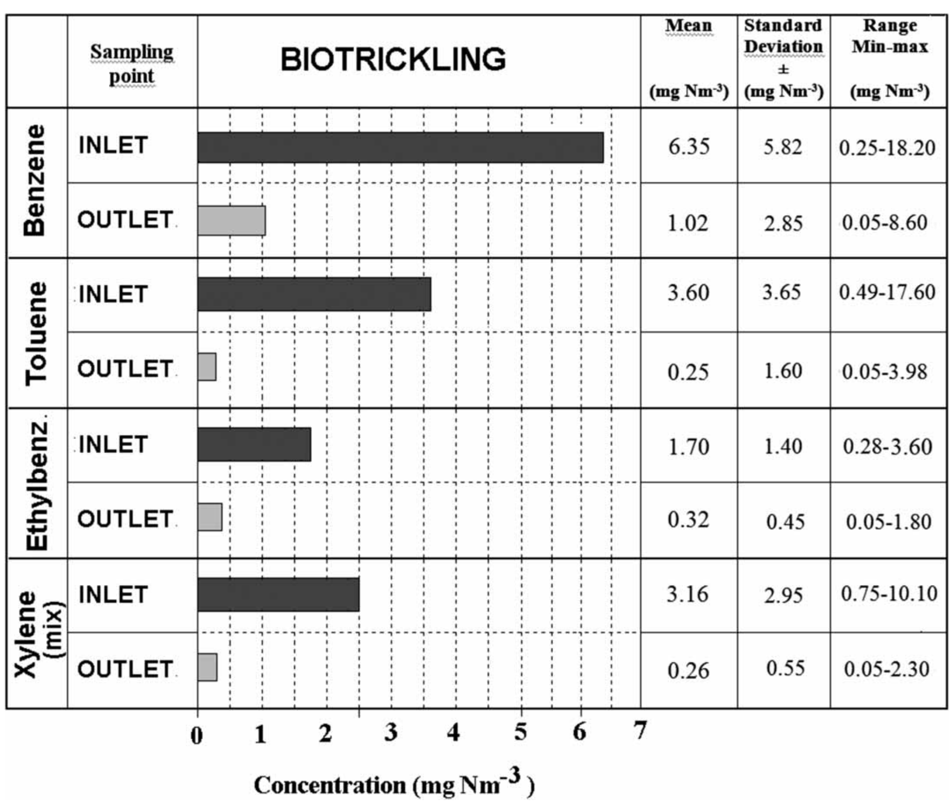


Figure 5. BTF performance for single BTEX removal. Bars represent the average value, whereas the table reports: mean (*m*); standard deviation (sd); range (min–max).

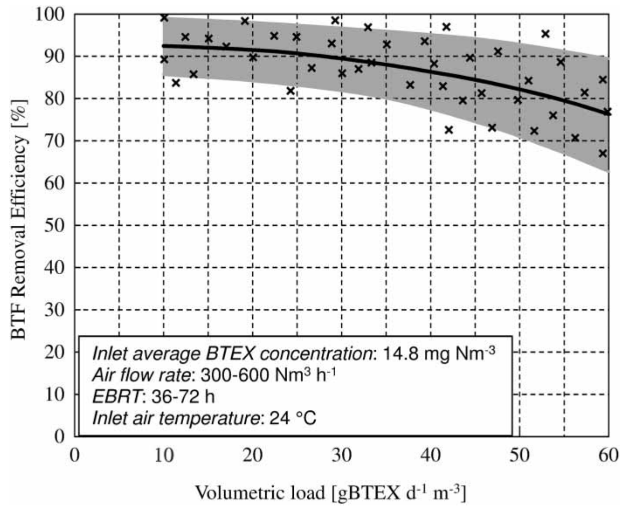


Figure 6. BTEX removal efficiency in the BTF as a function of the volumetric load referred to the total BTEX. The continuous line and the shaded area represent the mean and the 95% confidence interval, respectively.

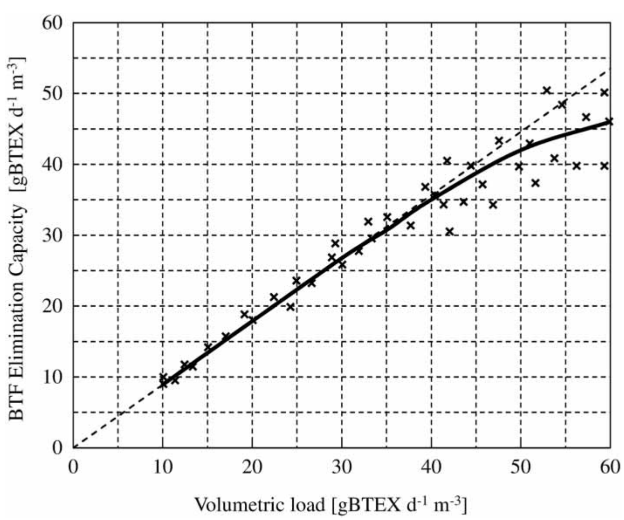


Figure 7. BTEX elimination capacity in the BTF as a function of the volumetric load. The thick continuous line is the interpolating curve, whereas the thin line shows a virtual linear behaviour.

are much lower than those found in other studies [34–40] which operated at higher inlet concentrations and higher volumetric loads.

Whole pilot plant performance

The whole two-stage experimented process has determined a 94.8% average removal efficiency for total BTEX. The average efficiency of the single compound has been always above 89% (benzene: 91.4%; toluene: 97.8%; ethylbenzene: 89.0% and total xylene: 97.3%). Considering the contribution of the single stages, an important amount of the average removal efficiency (58.6%) has been due to the scrubber, which has an elimination capacity equal to 75.6 g BTEX d^{-1} m^{-3}. The BTF contributed with an elimination capacity equal to 31.1 g BTEX d^{-1} m^{-3} (removal efficiency: 36.2%). Such a low value is due to the polishing role of the second stage.

These results are indeed very interesting even if they confirm the lower bioavailability of benzene and ethylbenzene with respect to toluene and xylene. However, very low average concentrations resulted in the final air effluent of the pilot plant: 1.02 mg Nm^{-3} for benzene; 0.25 mg Nm^{-3} for toluene; 0.32 mg Nm^{-3} for ethylbenzene and 0.26 mg Nm^{-3} for total xylene. Noticeable peak concentrations, albeit isolated, were detected (benzene: 8.60 mg Nm^{-3}; toluene: 3.98 mg Nm^{-3}; ethylbenzene: 1.80 mg Nm^{-3} and xylene 2.30 mg Nm^{-3}).

By comparing this result with the threshold concentrations of toxicological parameters (Table 1), it can be argued that only the peak concentrations of benzene may represent some health concern, as they are very close to the limits of the parameters STEL and TWA. A similar observation can be made for toluene and xylene, as regards the risk of odorous emissions.

It is reasonable to believe that the application of lower volumetric loads to the biological process may produce additional benefits on reducing these contaminants.

Conclusions

The experimentation carried out on a two-stage pilot plant, based on a pre-treatment with water scrubber and a final polishing BTF, has proved the effectiveness of BTEX removal from the waste air steam of a refinery WWTP. The air waste fed to the pilot plant was characterized by a very high variability of the inlet contaminant concentrations (benzene: 1.42–56.80 mg Nm^{-3}; toluene: 1.67–55.00 mg Nm^{-3}; ethylbenzene: 1.33–4.30 mg Nm^{-3} and xylene: 0.65–15.50 mg Nm^{-3}) around the average concentrations of 11.80 mg Nm^{-3} for benzene, 11.50 mg Nm^{-3} for toluene, 2.90 mg Nm^{-3} for ethylbenzene and 9.60 mg Nm^{-3} for xylene. The scrubber has proved to be very effective in the rough removal of the peak concentrations, such as to determine an average removal efficiency of 58.6% for total BTEX, in correspondence with an air/water ratio of 200 Nm^3 m^{-3}. This result was partly determined by biological degradation, as a noticeable biofilm growth has been observed on the packing surface.

The passage through the biotrickling stage has determined the additional removal efficiency of 87.5% for total BTEX. However, the experimentation has confirmed the better biodegradability of toluene and xylene with respect to benzene and ethylbenzene.

Overall, the whole two-stage process has determined a 94.8% removal efficiency of total BTEX, leading to outlet average concentrations of 1.02 mg Nm^{-3} for benzene,

$0.25\,\mathrm{mg\,Nm^{-3}}$ for toluene, $0.32\,\mathrm{mg\,Nm^{-3}}$ for ethylbenzene and $0.26\,\mathrm{mg\,Nm^{-3}}$ for xylene. By comparing these results with the threshold concentrations of toxicological parameters, such as TWA and STEL, it can be argued that only isolated peak concentrations of benzene may have some health concern. A similar observation can be made for toluene and xylene, as regards the risk of odorous emissions. Anyway, it is reasonable to believe that the application of lower volumetric loads to the biological process will lead to additional benefits on the removal of BTEX.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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