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Analytical Monitoring of the Chemicals Present in the Discharge Water Generated by the Surface Treatment Industry

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ABSTRACT

Industrial discharge water, and especially that from the surface treatment industry (ST), contains non-negligible amounts of pollutants even though the legislation is fully respected. In spite of this, no detailed studies list the exact chemical composition of these effluents. The present study reports the results of analyses performed over a 6-month period involving 15 standard water parameters. Over 160 substances including 33 metals, 58 volatile organic compounds (VOCs), 16 polycyclic aromatic hydrocarbons (PAHs), 24 chlorophenols (CPs), 16 alkylphenols (APs), 5 chloroanilines (CAs) and 7 polychlorobiphenyls (PCBs) were monitored. The industrial effluents presented polycontamination involving metals, minerals and organics with a high degree of qualitative and quantitative variability. Of the 160 substances monitored, 46 were regularly found: 25 inorganics including 8 metals (Co, Cr, Cu, Fe, Ni, Pb, Sn, Zn) and 21 organics (4 PAHs, 10 VOCs, 4 CPs and 3 APs). Eighteen were systematically presented at quantifiable levels.

Keywords: Discharge Water; Metal-Finishing Industry; Hazardous Substances; Analytical Monitoring

1. Introduction

In 1976, European legislation classified chemicals into two main lists of priority substances or groups of priority substances (Directive 76/464/EEC of 4 May 1976). The first called "The Black List" or "List I" concerns particularly dangerous substances such as hexachlorobenzene, pentachlorophenol, trichloromethane, mercury and cadmium, substances considered to be highly toxic, persistent and bio-accumulative. The second list *i.e.* "List II" or "The Grey List" gathers priority substances, such as benzene, dichloromethane, lead and nickel, which are considered to be less toxic than those of List I but nevertheless to present a significant risk to the environment [1-4]. These lists were then completed, following the Water Framework Directive of 2000 (Directive 2000/60/EC, WFD 23 October 2000) which aims to achieve good chemical and ecological status of all water bodies in Europe [1-3]. The WFD focuses in particular on ensuring a reduction of the volumes of effluent discharged and on monitoring target substances for which objectives of reduction and/or elimination were laid down to occur over a defined period of time. Currently in France, the list of substances included in the characterization of good chemical status includes 41 substances classified into three broad categories. The first concerns 13 substances blacklisted by the WFD including for instance 4-p-nonylphenol, anthracene, mercury and cadmium, for which the French target is a 50% reduction in releases by 2015 and the European target, elimination of discharges by November 2021 or December 2028. The second category covers 20 WFD priority substances, such as naphthalene, trichlorobenzene, octylphenols, dichloromethane, lead and nickel; the national objective for them is a 30% reduction in their release by 2015 while no deadline has been fixed at the European level. The other eight substances such as trichlorethylene and carbon tetrachloride were on list I of the 1976 directive, but are not included in the current WFD; the French objective for these molecules is a 50% reduction in discharges by 2015.

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Note that there is also another category which includes 139 substances on list II of 1976, including, for example, toluene, 1,2-dichlorobenzene, copper and zinc, which are also concerned by a national reduction plan (10% reduction by 2015).

The surface treatment (ST) industry is particularly affected by many of the chemicals mentioned [5-9]. Indeed, among industrial activities as a whole, ST is currently considered to be one of the sectors that consumes the most water and chemicals, and therefore one of the largest producers of wastewater, although a huge effort has been made in water treatment for over 20 years by ST plants [8,9]. To treat their wastewater, the ST sector generally uses physico-chemical methods, making various substances insoluble then separating the purified water from the sludge formed [10,11]. However, it is known that this decontamination technique cannot eliminate all pollutants. Even though it complies with the regulations in force, ST effluent contains significant mineral and organic polycontamination. Currently, Europe asks manufacturers to innovate to reduce and/or eliminate any chemicals present in their wastewater. However, before any actions can be taken to move towards zero pollution discharge, it is necessary to identify all the pollutants in the effluents qualitatively and quantitatively [2,3,10,11]. But, to our knowledge, there is no literature on this issue, in particular concerning the cocktails of chemicals present in the discharge water from the ST industry.

In the present study, we report the results of analytical monitoring of the chemical composition of the water discharged from a surface treatment company. To do this, we collected effluents characteristic of the industrial activity over six consecutive months, measured several parameters of the water and identified the presence of numerous chemicals to characterize the effluents.

2. Materials and Methods

2.1. Sampling

Wastewater was taken at the outflow from Galvanoplast Co Ltd located in Les Aynans (Haute-Saône, France). Galvanoplast specialises in galvanisation, phosphatation, electrophoretic painting and flake coating, all processes designed to protect metal parts against corrosion. To process its waste water, the company uses a physico-chemical treatment composed of three main steps: 1) pretreatment for hexavalent chrome, 2) treatment by chemical precipitation of the whole range of metal pollutants and 3) flocculation-decantation to separate the sludge formed from the water (defined as the wastewater) which then flows into the river if it satisfies legal requirements. Effluent outflow rate averaged ~8 m^3/h. Each sample of wastewater was representative of a whole day's activity. We sampled one outflow per month over 6 consecutive months. The three main concerns for the environment arise from the levels of zinc, nickel and COD (chemical oxygen demand) which are not far from the authorized limit.

2.2. Chemical Analysis

Full chemical characterisation of the wastewater was carried out by measuring several conventional parameters of the water and by looking for the presence of various chemicals. Thus, we measured 15 parameters and checked for the presence of 160 chemicals in the 6 samples. The water parameters monitored were: pH, conductivity, suspended solids (SS), COD, biological oxygen demand (BOD), total organic carbon (TOC), the levels of the different forms of nitrogen (nitrates, nitrites, ammonium nitrogen and Kjeldahl nitrogen), adsorbable halogenated organic compounds (AOX), the hydrocarbon index, free chlorine, and cyanides (total CN and easily released CN). In order to identify the inorganic and organic pollutants present in the wastewater, we analysed eight broad categories of substances classified as follows: inorganic compounds including metals (33 metal ions), volatile organic compounds (VOCs, 58 molecules), polycyclic aromatic hydrocarbons (PAHs, 16 molecules), chlorophenols (CPs, 24 molecules) alkylphenols (APs, 16 molecules), chloroanilines (CAs, 5 molecules), polychlorobiphenyls (PCBs, 7 molecules) and other substances (4 organo-tin complexes, and a few other molecules). The experimental protocols have already been detailed elsewhere [10,11]. The COD was assessed following protocol ISO 15705 which requires oxidation by potassium bichromate in hot acid medium (150˚C) for 2 hours. The equipment used was the Vario COD measurement system (Aqualytic, Dortmund, Germany) with Eco8 reactors from Velp Scientifica (Milan Italy). The 5-day BOD protocol involved measurement of the initial concentration of O_2 initially dissolved in the sample (C_0) and the concentration of O_2 after five days of incubation in the darkness at 20˚C (C_s). The BOD_5 was thus equal to C_0 - C_5 and is expressed in mg of O_2/L. We used the so-called respiration method following standard NF EN 1899-2 which measures the consumption of O_2 through the drop in pressure in the vials by means of a manometer and a KOH trap to absorb the CO_2 evolved through respiration. The method is simple, reproducible and reliable (no problems of dilution) but is rather time-consuming (5-day incubation). The measurements were made with an Oxi-Direct system and an ET 618-4 incubator (both from Aqualytic, Dortmund, Germany). Metal analysis was performed by ICP-AES (ThermoFisher, iCAP 6500 radial model, Courtaboeuf, France) fitted with a rapid sampling loop (FAST). An internal standard, Yttrium, was added continuously in order to overcome any variation in the response of the instrument during analysis. Analysis accuracy was estimated using certified references (Hard Drinking Water, ERM-CA011, LGC Promochem, Molsheim, France). Quality control (half a point on the calibration scale prepared independently of the calibration) was inserted every 10 samples to check for the absence of drift. The concentrations of other ions were measured by ionic chromatography (orthophosphates, nitrites) or by photometry (F, B) following standard French protocols (NF EN ISO 10304 for anions). The VOCs (NF EN ISO 15680), PAHs (MET 72G), CPs (NF EN 12673), AOX (NF EN ISO 9562), CAs (MET INT), hydrocarbon index (NF EN ISO9377) and APs (MET INT) were analysed by an accredited analysis laboratory (LCDI, Marange, France). The results are expressed in mg/L or µg/L.

3. Results

Which parameters and pollutants to monitor? The choice of parameters and substances was determined by the regulatory requirements specifically laid down for the surface finishing plant (as stipulated in the French law of 5 September 2006). The company must monitor a clearly defined set of parameters and substances for which limit levels have been fixed by county law (**Table 1**). The company must permanently record the volume of water discharged and the pH, and also make a daily records of the temperature and the levels of chromium VI, nickel, zinc and easily released cyanides. Then, once a week, it must follow its concentrations of chromium III, copper and iron. Finally, every quarter, an accredited laboratory must determine Cr(VI), Cr(III), Cu, Fe, Ni, Zn, P, F, SS, N_{TOT}, CN, COD, nitrites, the hydrocarbon index and the AOX. Note that no organics are mentioned in this list. However, following the recommendations of 5 January 2009 concerning dangerous substances in water, the ST companies must monitor new target molecules for the ST industry including nonylphenols, fluoranthene, naphthalene, anthracene, trichloroethylene, tetrachloroethylene, dichloromethane, carbon tetrachloride and toluene.

Table 1. Maximum authorized levels of emission in treated industrial discharge water, expressed in average concentrations.

Parameter or pollutant	Average daily concentration	Analysis frequency
pH	6.5 - 9	continuous
Zinc	3000 µg/L	daily
Nickel	2000 µg/L	daily
Iron	5000 µg/L	weekly
Copper	2000 µg/L	weekly
Chromium III	2000 µg/L	weekly
Chromium VI	100 µg/L	daily
Easily released CN	0.1 mg/L	daily
Nitrites	20 mg/L	three-monthly
Total nitrogen	50 mg N/L	three-monthly
P	10 mg/L	three-monthly
COD	300 mg/L	three-monthly
SS	30 mg/L	three-monthly
Hydrocarbon index	5 mg/L	three-monthly
AOX	5000 µg Cl/L	three-monthly

3.1. Water Parameters

Table 2 shows the results obtained for the 15 water parameters measured in the six samples. The discharge water samples were slightly alkaline (mean pH of 8.04 at 20˚C). They did not present significant turbidity (low levels of SS) and were not biodegradable (very low BOD). On the other hand, they showed high conductivity (mean value 5969 ± 386 µS/cm) and relatively high levels of organic pollution with a mean COD of 198 ± 26 mg/L and TOC of 48.7 ± 6.3 mg/L. High levels can also be seen in total nitrogen (71.7 ± 4.8 mg/L) and AOX (1020 ± 200 µg Cl/L). The hydrocarbon index, cyanides (total and easily released) and free chlorine were lower than the quantification limit (**Table 2**).

3.2. Inorganic Substances

The results reported in **Table 3** confirm the high salinity of the discharge water with high levels of chloride ion (mean value 1650 ± 339 mg/L), sulphates (258 ± 16 mg/L), calcium (175 ± 13 mg/L) and sodium (1057 ± 116 mg/L). These results are also in agreement with the issues faced by the company concerning the levels of zinc and nickel, occurring at high levels (mean values 874 ± 98 µg/L and 408 ± 111 µg/L respectively), even though they remain below the authorized limits (**Table 1**). Ten other metals were also found at quantifiable levels (**Table 3**): Li, Mn, Mo, Se, Co, Cr, Cu, Fe, Pb and Sn. The concentration of other metals remained below the limit of quantification (Al, Ba, Cd, Sr, Ti and V), or below the limit of detection (Sb, As, Cr VI, Hg, Pd, Pt, Ag, Te, Tl, W).

3.3. Organic Substances

Twenty-one organic substances were identified and quantified including certain VOCs, PAHs, CPs and APs (**Table 4**). In the first category, only chloroform was systematically found in all six effluent samples. All the other substances, in particular chloroanilines and poly-

Table 2. Parameters for the six samples of industrial discharge water (DW).

Parameter	Unit	QL[a]	DW 1	DW 2	DW 3	DW 4	DW 5	DW 6
pH (20˚C)			8.05	7.99	8.1	8.0	7.9	8.2
Conductivity (20˚C)	µS/cm	10	5250	6351	5974	6021	6250	5970
SS	mg/L	2	17	21	15	19	24	22
TOC	mg/L	0.5	45.5	39.3	49.2	41.7	35.2	51.9
COD	mg/L	5	177	163	202	194	215	235
BOD	mg/L	3	37	42	45	39	51	58
Total nitrogen	mg N/L		70.9	63.9	77.3	73.2	69.3	75.5
Nitrates	mg/L	0.1	210	157	192	206	195	178
Nitrites	mg/L	0.1	21	13.2	16.8	24.5	17	20
Kjeldahl nitrogen	mg N/L	1	17.1	24.5	28.9	19.3	20.1	29.3
Ammonium nitrogen	mg/L	0.05	0.181	0.23	0.31	0.19	0.25	0.2
Total CN	mg/L	0.02	<QL	<QL	<QL	<QL	<QL	<QL
Easily released CN	mg/L	0.02	<QL	<QL	<QL	<QL	<QL	<QL
Free chlorine	mg/L	0.02	<QL	<QL	<QL	<QL	<QL	<QL
AOX	µg Cl/L	10	730	1023	986	958	1075	1350
Hydrocarbon index	mg/L	0.1	<QL	<QL	<QL	<QL	<QL	<QL

[a]Quantification limit.

chlorobiphenyls were below the limit of quantification.

4. Discussion

The analytical characterisation of the six samples of discharge water showed that the physico-chemical treatment used by the company to decontaminate its wastewater did not enable total abatement of all the pollution. The samples were found to still contain mineral pollution (high conductivity and salinity), an organic load presenting high values of COD and TOC, and a mixture of metals and organics that was not negligible. However, the legal requirements were satisfied. It can also be noted that the pollutant load varied over time both quantitatively and qualitatively.

The 6-month sampling campaign identified and quantified a total of 46 chemicals (**Table 5**) that can be grouped into a series of 25 inorganics including 8 metals (Co, Cr, Cu, Fe, Ni, Pb, Sn, Zn) and 21 organic pollutants (4 PAHs, 10 VOCs, 4 CPs and 3 APs). Of the 46 substances, 18 were systematically at quantifiable levels (Cr, Fe, Ni, Zn, Mn Mo, Se, Li, P, Si, Na, K, Mg, Ca, SO_4, Cl, F and chloroform). Note that the concentrations found ranged from a few g/L for minerals to a few µg/L (traces) for organics with intermediate levels of mg/L for metals. However, if these same values are expressed as quantities discharged rather than concentrations, some quite high values are reached, especially for minerals. For instance, for the three main issues that the company has to face (COD, Zn and Ni), the mean concentrations in the six discharge waters were 198 ± 26 mg/L for the COD (38 kg/day), 0.87 ± 0.1 mg/L for zinc (*i.e.* 168 g/day) and 0.41 ± 0.11 mg/L for nickel (78 g/day). Also, the values varied over time (RSDs of 13, 11 and 27% for COD, Zn and Ni, respectively). Moreover, this variability over the whole 6 months of the study is also found over shorter periods, as seen in **Figure 1** which reports the levels of COD, Zn and Ni over 54 hours of monitoring with RSDs of 14, 11 and 19% respectively. This observation is confirmed by the results in **Figure 2** which describes the variations in the concentrations of the parameter AOX (RSD = 69%) and the substance chloroform (RSD = 85%) over 45 days (9 samples). This illus-

Table 3. Concentrations of the inorganic substances present in the six samples of industrial discharge water.

Ion	Unit	QL[a]	DW 1	DW 2	DW 3	DW 4	DW 5	DW 6
Chlorides	mg/L	0.1	1200	2081	1766	1450	1450	1955
Fluorides	mg/L	0.1	3.8	5.9	6.17	7.2	2	5.5
Sulphates	mg/L	0.1	260	239	247	273	250	280
Calcium	mg/L	0.15	169	193	171	184	155	175
Magnesium	mg/L	0.002	7.02	8.9	6.9	7.9	7.5	6.5
Potassium	mg/L	0.075	59.2	62.5	77	69	70	58
Sodium	mg/L	0.019	907	1041	1229	1153	990	1020
Silicon	mg/L	0.011	2.45	2.9	2.7	1.9	2.5	2.1
Bromides	mg/L	0.1	<QL	<QL	<QL	<QL	<QL	<QL
Carbonates	mg/L	0.02	<QL	<QL	<QL	<QL	<QL	<QL
Phosphorus	mg/L	0.007	0.064	0.23	0.45	0.39	0.15	0.35
Orthophosphates	mg/L	0.1	<QL	<QL	<QL	0.21	<QL	0.13
Tributylphosphate	µg/L	0.02	<QL	<QL	<QL	0.11	<QL	0.31
Lithium	µg/L	10	15	21	30	20	18	20
Manganese	µg/L	0.6	35	41	50	40	30	45
Molybdenum	µg/L	10	24	30	31	10	20	28
Selenium	µg/L	11	20	20	20	20	<QL	<QL
Cobalt	µg/L	10	630	778	555	712	647	519
Chromium	µg/L	3	929	1020	850	1090	950	888
Chromium VI	µg/L	0.01	<QL	<QL	<QL	<QL	<QL	<QL
Copper	µg/L	4	<QL	40	<QL	70	<QL	33
Iron	µg/L	3	838	1150	1030	950	790	877
Nickel	µg/L	2	270	389	610	370	410	396
Lead	µg/L	8.5	<QL	<QL	21	30	<QL	25
Tin	µg/L	7	<QL	90	80	100	<QL	<QL
Zinc	µg/L	0.4	758	810	982	811	888	995

[a]Quantification limit.

trates how the RSDs over a period of 6 months are similar or even lower (RSDs of 20% and 94% for AOX ··· and than those over a shorter time. So from the overall results, it can be noted that the variability over 6 months is simply the result of variability over a much shorter time scale. The daily and monthly fluctuations noted are not easily controllable as they depend not only on the variety of industrial processes carried out (different parts to be treated, multiple treatments, etc.) but also on dysfunctions liable to occur in the waste water treatment

Table 4. Concentrations of organic substances in the six samples of discharge water.

Molecule	Unit	QL[a]	DW 1	DW 2	DW 3	DW 4	DW 5	DW 6
2-Chlorophenol	µg/L	0.01	<QL	3.89	5.23	4.57	<QL	<QL
4-Chlorophenol	µg/L	0.01	<QL	0.91	1.8	1.34	<QL	<QL
2,4-Dichlorophenol	µg/L	0.01	<QL	2.33	1.4	1.99	<QL	<QL
2,4,6-Trichlorophenol	µg/L	0.01	<QL	2.01	1	1.76	<QL	<QL
Nonylphenol	µg/L	0.1	0.51	0.23	0.11	0.29	0.36	<QL
Nonylphenol diethoxylate	µg/L	0.1	1.13	0.27	0.76	1.23	0.49	<QL
Octylphenol diethoxylate	µg/L	0.1	18.5	3.95	6.7	4.91	5.1	<QL
Chloroform	µg/L	1	49	11	37	151	9	123
1,2-Dichloroethane	µg/L	0.2	0.99	2.2	3.8	2.9	<QL	<QL
Dichloromethane	µg/L	1	1.3	3.4	4.4	1.5	<QL	<QL
Tetrachlomethane	µg/L	0.2	<QL	1.3	<QL	1.1	<QL	<QL
Tetrachloroethylene	µg/L	0.2	<QL	97	<QL	<QL	<QL	<QL
Trichloroethylene	µg/L	0.2	<QL	1.9	<QL	<QL	<QL	1.3
1,2-Dichlorobenzene	µg/L	0.2	79	286	159	179	<QL	<QL
Toluene	µg/L	0.2	<QL	1.4	1	1.2	<QL	1.9
Ethylbenzene	µg/L	0.2	<QL	3.9	<QL	1.6	<QL	<QL
Isopropylbenzene	µg/L	0.2	<QL	<QL	3.9	2	<QL	<QL
Naphthalene	µg/L	0.01	0.034	0.14	0.09	0.12	0.21	<QL
Phenanthrene	µg/L	0.01	<QL	0.033	0.06	0.045	<QL	<QL
Anthracene	µg/L	0.01	<QL	0.02	<QL	<QL	<QL	0.08
Fluoranthene	µg/L	0.01	<QL	0.011	<QL	<QL	<QL	<QL

[a]Quantification Limit.

Table 5. Recap of the total number of substances identified in the whole set of 6 discharge water samples and the total number of substances per sample.

Group of substances	Number of substances analysed	DW 1	DW 2	DW 3	DW 4	DW 5	DW 6
Metals	8	5	7	7	8	5	7
VOCs	10	4	9	6	8	1	3
PAHs	4	1	4	2	2	1	1
CPs	4	0	4	4	4	0	0
APs	3	3	3	3	3	3	0
Other inorganic pollutants	13	13	13	13	13	12	12
Other pollutants[a]	4	2	2	2	4	2	4
Total	46	28	42	37	42	24	27

[a]Nitrates, nitrites, orthophosphates, tributylphosphate.

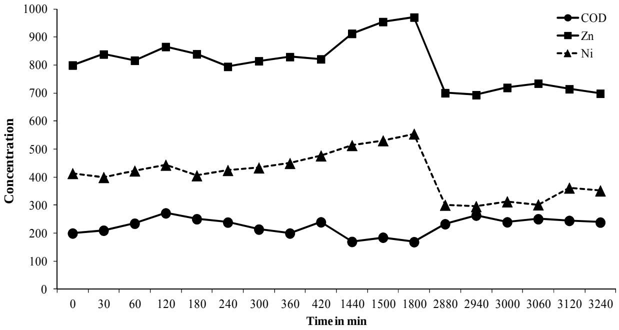

Figure 1. Variation of the levels of COD, Zn and Ni in mg/L over 54 hours of analytical monitoring.

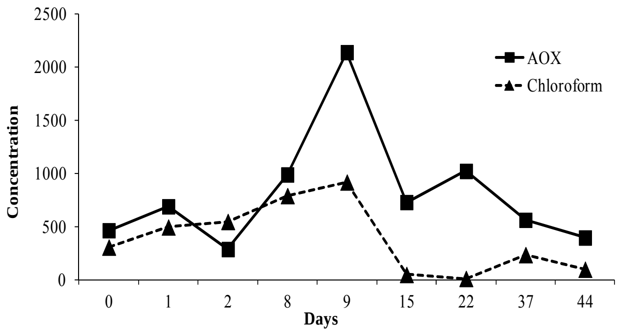

Figure 2. Variation of concentrations of AOX and chloroform in µg/L over 45 days.

plant. The high variability of the levels of pollution arriving in the treatment plant is not taken into account in the continuous decontamination of the wastewater, which could explain the variations that were noted in the samples taken at the outlet of the plant.

The number of substances found in the discharge water was also highly variable. The minimum was 24 substances found in DW 5 while this value was doubled in DW 2 and DW 4 (**Table 5**). Finally, we can consider that our figure of 46 chemicals present, is an underestimation since some substances (Al, Ba, B, Sr, Ti, V, CAs, PAHs, organo-tin) were identified but not quantified. Moreover, other organic substances (chloronitrobenzenes, chloronitrotoluenes, chlorotoluidines, etc.) were not assayed for financial or analytical reasons (techniques not available or not sufficiently sensitive), although it is known that they could potentially be present in the discharge water owing to the use of numerous complex cleaning agents in the industrial processes, their exact composition often being difficult to ascertain.

5. Conclusion

This study has shown that although the effluent produced by the surface treatment company studied here respects the authorized discharge levels, it does contain a whole cocktail of chemicals. They lead to relatively strong polycontamination with metals, minerals and organic compounds. The polycontamination is variable in time both qualitatively and quantitatively. In all, 46 substances were identified and quantified including 8 metals and 21 organics. Of the 46, 18 were systematically found including zinc and nickel, two substances that the industry has problems eliminating. It was also shown that the variations in the levels and parameters studied over the 6 months of the investigation were similar to those noted over much shorter periods of time. The variations were attributed to the differences in activities carried out by the company over time. Decontamination of the effluent was a continuous process and it was unable to systematically handle all the fluctuations in the pollutant levels at the plant's inlet. For the follow-up of this study, it would be interesting to identify the origin of the substances, especially organics, by means of analytical monitoring of the effluent at the inlet to the treatment plant and also how the levels of the pollutants change as the waste water flows through the plant.

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