

## PHYSICO-CHEMICAL DEGRADATION OF SOME LAND-FILL WASTES CONTAMINATING ON SURFACE AND GROUND SURROUNDING WATERS

Maria DIACONU<sup>1</sup>, Silvia GAROFALIDE<sup>1,2,\*</sup>, Georgiana COCEAN<sup>1,4</sup>, Alexandru COCEAN<sup>1,2</sup>, Daniela Angelica PRICOP<sup>1,3</sup>, Iuliana COCEAN<sup>1</sup> and Silviu GURLUI<sup>1,\*</sup>

<sup>1</sup> "Alexandru Ioan Cuza" University of Iași, Faculty of Physics, Atmosphere Optics, Spectroscopy and Laser Laboratory (LOASL), 11 Carol I Bld. 700506 Iași, Romania;

<sup>2</sup> "Alexandru Ioan Cuza" University of Iași, Research Center with Integrated Techniques for Atmospheric Aerosol Investigation in Romania, RECENT AIR, Laboratory of applied meteorology and climatology, A Building, Physics, 11 Carol I, 700506 Iași, Romania

<sup>3</sup> "Alexandru Ioan Cuza" University of Iași, Research Center with Integrated Techniques for Atmospheric Aerosol Investigation in Romania, RECENT AIR, Laboratory of Astronomy and Astrophysics, Astro-nomical Observatory, Physics, 11 Carol I, 700506 Iași, Romania

<sup>4</sup> Rehabilitation Hospital Borșa, 1 Floare de Colt Street, 435200 Borșa, Romania

### Abstract

*Influences of landfills on the surrounding waters have been studied in the neighbourhood of an urban area. Analytical and spectroscopic methods were used to investigate the waters of a canal situated in the neighbourhood of the landfill. Dangerous chemical compounds have been identified in the water as pollutants resulting from the physico-chemical processes of degradation of landfill waste. The quick method with gas sensor detection and colorimetric ion identification using a commercial Quantofix kit, as well as precipitation with AgNO<sub>3</sub>, offered preliminary information completed by Fourier Transform Infrared Spectroscopy (FTIR). Pollutants that reached the canal waters either by air (evaporation) or by discharges or through rainwater were analysed from the point of view of the degree of danger, sources of waste materials, as well as their circuit in the environment. Hydrogen cyanide, nitrites, nitro compounds, sulfites and sulphates, sulfonic tension-active compounds, peroxydes and hyperoxydes, azo-compounds, organic and inorganic acids of high corrosive potential, and Fe ions were generally the detected pollutants, each of them with specific harmful potential contributing as well in a cumulative effect to endangering human health directly or indirectly as environmental circulating contaminants.*

**Keywords:** Landfill wastes; Leachate; Hydrogen cyanide; FTIR of water pollutants

### Introduction

The problems caused by environmental pollution are increasing due to the effects on the quality of life, from the discomfort caused by odors and unpleasant appearances caused by the waste dumped in landfills to the endangerment of human health induced by chemical compounds formed by partial decomposition or degradation of waste resulting from household and vegetable sources [1, 2]. Leachate resulting from waste decomposition is a source of pollutants such as dissolved organics, inorganic macrocomponents, heavy metals, and xenobiotic organic compounds that can spread in the environment, affect the ecosystem, enter the food chain, and impact human health [3, 4]. The environmental pollution induced by

\* Corresponding author: sgurlui@uaic.ro and silvia.garofalide90@gmail.com

landfills into underground and surface water, including the ocean, and also in the atmosphere due to the suspension of particles and outdoor pollution has been studied due to its carcinogenic and non-carcinogenic effects on the exposed population living in their vicinity [5-8]. Landfill gas emissions such as ammonia, sulphides, methane, and carbon dioxide result in odor pollution involving toxic and fire risks [9, 10]. Studies of leachate characteristics—the liquid part of the waste—using elaborated methods showed components of polluting potential for the surrounding environment [11], and treatment techniques for the leachate have been proposed [12].

All this research activity, having as a common point the danger posed by landfills, shows that greater attention must be paid to the monitoring of the areas in their vicinity, both from the point of view of air quality and from the point of view of leachate compounds. Moreover, stopping the storage activity in landfills as a result of their closure without continuous measures to carry out specific purification, filtering, decontamination, and neutralisation processes can lead to contamination of the atmosphere and air in the neighbouring region. Thus, by stopping the leachate treatment facilities, it leads to their possible discharge into the surface and underground waters of the area, contaminating these waters. From this perspective, we propose that through this work we highlight methods that can be practically applied for an immediate evaluation and easy and low-cost monitoring at frequent intervals so that decontamination measures can be taken in a timely manner.

## Experimental part

### *Materials*

#### *Studied area*

In the context of the complaints of the inhabitants of an urban area regarding the pollution noticed in the vicinity of a closed landfill situated in the neighbourhood of the city, water samples were taken from a ditch located in its immediate vicinity. The analysis of the waters in the vicinity of landfills provides valuable information on the contamination induced by them in the area, both in water, soil, and air [1, 13]. Being a ditch that accumulates leachate, which is mainly the result of rainwater that "washed" the garbage, it reflects compounds that also spread into the soil and groundwater [1-3]. The rainwater collected in the ditch also accumulated compounds from the particles dispersed in the atmosphere [10, 13, 14] as a result of the physico-chemical degradation processes of wastes stored in the landfill under the action of light, heat, biological agents, partial combustion, etc.

#### *Physicochemical analyses*

A Quantofix kit containing semi-quantitative test strips for water analysis acquired from Macherey-Nagel, Germany, was used for an analytical quick test to identify water hardness #91201 in the range 0 – 25<sup>o</sup>d (10d ~5.555mol/m<sup>3</sup>), carbonate hardness #913 23 in the range of 3 – 200d (1<sup>o</sup>d = 17.8 mg/L CaCO<sub>3</sub>), SO<sub>4</sub><sup>2-</sup> ions (#913 29) in the range of 200 – 1600mg/L, SO<sub>3</sub><sup>2-</sup> ions (#913 06) in the range of 10 – 1000mg/L, NO<sub>3</sub><sup>-</sup> and NO<sub>2</sub><sup>-</sup> (#913 13) in the range of 10 – 500mg/L and 1 – 80mg/L, respectively, ions of Fe<sup>2+</sup>/Fe<sup>3+</sup> ions (#913 44), in the range of 2 – 100mg/L.

The instructions for using the Quantofix kit are provided by the producer, printed on paper and as pictograms on the containers (Supplementary Materials, Figure 1a). The basic procedure for the tests used herein consists of dipping the test strip into the aqueous sample for a time *td*, shaking off the excess liquid, and comparing the test field with the colour scale after the time indicated in the instructions (reading time, *tr*). The dipping time (*td*) and the reading time (*tr*) are presented in the Supplementary Materials, Table 1, for each of the test strips used for this investigation. In the same Table 1 of the Supplementary Materials are also listed the interferences indicated by the producer. The pH value of the sample of water of 5.7 is acceptable for the accuracy of the results of the tests performed with the Quantofix kit, and no treatments of the sample prior to testing were required.

The pH was tested with the pH tester HI98103 from Hanna Instruments Inc., Woonsocket, Rhode Island, USA (Supplementary Materials, Figure 1b).

Cyanides were preliminary detected in the samples of water taken from the ditch in the neighbourhood of the landfill based on the precipitation test with silver nitrate ( $\text{AgNO}_3$ ) in a 2 mol/L dilute nitric acid solution and measuring airborne releases of hydrogen cyanide, HCN with a portable multi-gas detector custom produced by BOSEAN, Zhengzhou, Henan, China (Mainland), with a range of detection of 0 – 50ppm.

*FTIR analysis*

Fourier Transform Infrared Spectroscopy (FTIR) analysis was performed with the FT-IR Spectrometer Bomem MB154S spectrometer at an instrumental resolution of  $4\text{cm}^{-1}$  (Bomem, ABB Group, Canada) on the residual material that resulted after the water evaporated by drying at room temperature ( $200^\circ\text{C}$ ) [1, 13]. In this sense, drops of the sample of water in a total amount of 0.5mL were put on glass slides, and after drying, the deposited solid material was removed by scraping with a stainless-steel spatula. The solid sample resulting from the evaporation of the water sample was then incorporated into about 280mg of potassium bromide (KBr) used as suspension media. From the obtained mixture of sample and KBr, about 250–260mg were pressed into a stainless-steel ring of 12mm inner diameter under 100atm pressure using a pneumatic pressing device. The ring with the pellet was placed in the sample holder of the spectrometer.

**Results and discussion**

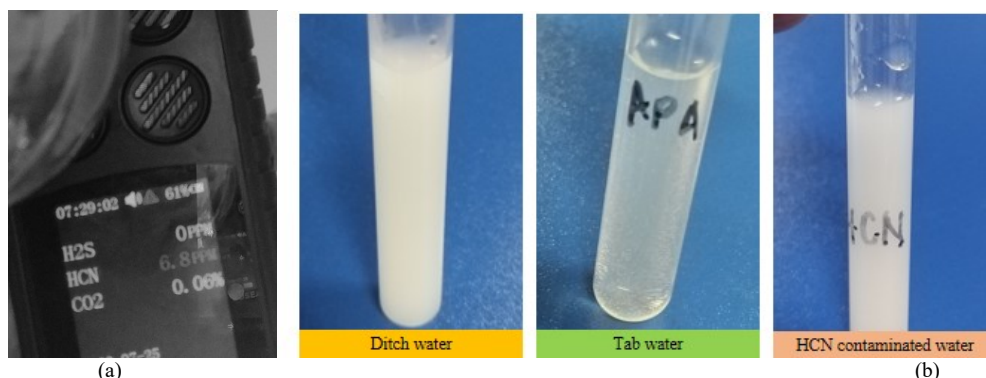
*Preliminary quick testing*

The preliminary quick testing and estimated chemical components of the water pollutants were performed with the aim of finding a method to immediately evaluate contamination risks when required.

**Table 1.** Rapid tests of HCN airborne and of pH.

Parameter	Detected/ Analysed Values	Values within normal limits	Evaluated quality
HCN gas	6ppm in air	Less than 3.3ppm/30min ( <i>J.E. Leiser et al.</i> , 1990 [15])	In excess (x2)
HCN in water	Heavy precipitation with $\text{AgNO}_3$ , milky white precipitate	Qualitative evaluation	CN groups identified
pH	5.7 (acid) Variable between 4.5 – 5.8, stabilizing after about 20 minutes at 5.7 as a result of continuous stirring	7 – 7.2	Corrosive

The slightly pink appearance of the water with white particles in suspension denotes anthropogenic-induced pollution. Detection of hydrogen cyanide released from a sample of water of 6ppm (Fig. 1a and Table 2) in the air exceeds a "normal limit" of 3.3ppm/30min as per *J.E. Leiser et al.*, 1990 [15] and National Research Council (US) Subcommittee on Acute Exposure Guideline Levels [16], which report that above the normal limit, HCN can induce dizziness, headache, and weakness, depending on the tolerance of each body. For detecting the presence of HCN in the water sample, the test was performed with silver nitrate in a 2mol/L dilute nitric acid solution. A milky white precipitate was thus obtained (Fig. 1b), which indicates the presence of cyan groups, possibly in  $[\text{Fe}(\text{CN})_6]^{4-}$  ions.



**Fig. 1.** The HCN detection with BOSEAN sensor (a) and the test with silver nitrate for CN groups detection in water (b)

A pH value of 5.7 (Table 2) was measured after stirring the sample of water for 20 minutes. Until the pH stabilised at 5.7 through a somewhat homogeneous distribution of the dispersion, it varied in the 20 minutes between values from 4.5 to 5.8. This pH value denotes water with corrosive potential on vegetation and metal constructions, as well as, by evaporation, a corrosive effect on the respiratory tract. For a correct estimation of the acidic character of the sample of water at this stage of preliminary analysis, the pH value must be correlated with the carbonate ions.

The results of the semi-quantitative tests were obtained by comparing the colour on each stripe with the closest matching colour on the container, which corresponds to a certain quantity, and were centralised in Table 2.

**Table 2.** Semi-quantitative measurements of the ions in the sample of water from the ditch in the landfill vicinity were performed with the Quantofix kit

Parameter	Detected/ Analysed Values	Values within normal limits	Evaluated quality
CO <sub>3</sub> <sup>2-</sup> ions (carbonate)	20 degrees kH hardness 436mg/L CaCO <sub>3</sub> equivalent	Hard water	Expected alkaline
NO <sub>3</sub> <sup>-</sup> ions (nitrate)/	10mg/L	10mg/L	At limit
NO <sub>2</sub> <sup>-</sup> ions (nitrite, also nitroderivatives)	1mg/L	0.05mg/l	In excess (x20)
SO <sub>4</sub> <sup>2-</sup> ions (sulfate)	<200mg/L	200mg/L	Normal
SO <sub>3</sub> <sup>2-</sup> ions (sulfite)	5mg/L	1 – 5mg/L	Acceptable
Fe <sup>2+/3+</sup> ions (iron)	5mg/L	0.4mg/L in water	In excess (x10)

The results of the stripe tests with the Quantoifix kit performed on the samples of water are presented in Table 2. Semi-quantitative analysis with Quantofix kH hardness test strips showed a high carbonate ion content, measured in degrees of hardness. Thus, the value of about 20<sup>0</sup>d, equivalent to 436mg/L calcium carbonate (CaCO<sub>3</sub>), would be more specific to alkaline water. The fact that this is not the case and the respective water has an acidic character indicated by its pH leads to the suspicion that other types of substances are present than carbonates, especially organic acids. The latter are not neutralised by the alkaline nature of the carbonates. Such organic acids may come from cleaning products used to wash carpets and toilets, including but not limited to formic acid, acetic acid, citric acid, and others. (If alkaline detergents are used for washing clothes, acidic cleaning products are used for washing carpets and toilets.) Another source may even be amino acids from bacteria, especially those that are difficult to mix with water (alanine, glutamine, etc.). Other compounds with an acidic character or which may change the pH by the transformations undergone (chemical reactions) in the

interaction with other compounds, as well as by exposure to high temperatures or under enzymatic action (vinegary of plant foods, especially fruits, juices, and others).

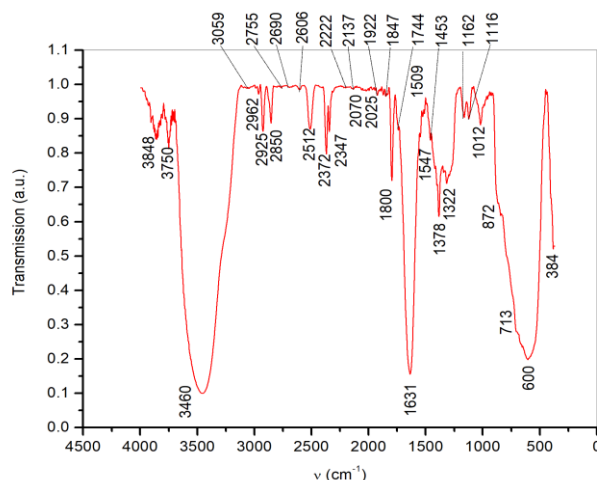
Quantofix semi-quantitative test strips for nitrate ions ( $\text{NO}_3^-$ ) and nitrite ions ( $\text{NO}_2^-$ ) show levels at the upper limit of 10mg/L nitrates and of 20 times more nitrites than the upper limit (1 mg/l measured compared to 0.05mg/L upper limit). Nitrate ions may contribute to acidic pH as nitric acid ( $\text{HNO}_3$ ). The group  $\text{NO}_2$  detected with Quantofix test strips can be assigned to nitrite ions ( $\text{NO}_2^-$ ), as well as organic nitroderivatives, including dyes with benzene structures, and this could explain the pinkish colour of the analysed sample of water where the nitro ( $-\text{NO}_2$ ) group may be involved in the chromophore structure.

Sulfate ions ( $\text{SO}_4^{2-}$ ) detected with semi-quantitative Quantofix test strips are within the normal limits of 200mg/L. Although their level is not worrying, they can contribute to the acidic nature of the pH through a cumulative effect with the other compounds. Acceptable levels are also found for sulfite ions ( $\text{SO}_3^{2-}$ ) with the specific test strips of Quantofix. Sulfite ions may also contribute to an acidic pH. They may also be part of the sulfonic groups of sulfated fatty acids or other organic sulfonic compounds.

Semi-quantitative Quantofix test strips for iron ions ( $\text{Fe}^{2+/3+}$ ) show 10 times exceedances of the limit values (5mg/L measured compared to 0.4mg/L limit admitted in water). Iron can be picked up due to corrosive compounds that have passed through metal pipes or come into contact with Fe-based metal objects.

#### ***Fourier Transform Infrared spectroscopy (FTIR)***

The FTIR spectrum (Fig. 2) of the dried material obtained from the sample of ditch water confirmed the results provided by the preliminary analyses and provided information on the organic components in the ditch waters that are from anthropogenic sources.



**Fig. 2.** FTIR spectra of residue collected after water evaporation

Vibration modes at 3460, 1631 and 1547 $\text{cm}^{-1}$  indicate  $\text{NH}_2$  and  $\text{NH}$  groups in amines, as well as in aromatic and heteroaromatic compounds like pyrrole, pyridine, imines, and oximes. Pyrrole, pyridines, imines, and oximes [1, 13, 17–20] may be the results of eventual combustion of plastic, dyestuffs, detergents, polyacrylonitrile, or leaves or their degradation under the sun's heat and enzymatic action [1]. In the same range, hydroxyl groups in carboxylic acids are assigned. Bands at 1631, 1744 and 1800 $\text{cm}^{-1}$  were assigned to carbonyl groups in amides (sourced in wastes of polyamides, nylon, etc.), amino acids (animal waste, processed meat, etc.), but also to carboxylic acids [1, 13, 20]. Carboxylic acids contribute to the acidic pH of the water and can come from products for washing carpets or result from vinegaring household waste (juices, fruits, etc.). The corrosive action can lead not only to the deterioration of iron

constructions but, through the release of CO<sub>2</sub> and even methane after decomposition under the action of heat and other compounds, they bring a substantial contribution to air pollution, sometimes with flammable potential. Peroxides and hyperoxides were identified in the bands of 3460, 1162 and 1116cm<sup>-1</sup> [17], and the source of contamination can be in household cleaning products, including detergents, as a result of their discharge in the form of packaging containing residues or in other forms. Carbonates are denoted by the 1453cm<sup>-1</sup> peak, indicating detergents and other cleaning products. Bands at 2925, 1378, 1162, 1116 and 872cm<sup>-1</sup> are assigned to the SO group in sulfonic acids (R-SO<sub>2</sub>-OH/RO-SO<sub>3</sub>-), sulfones, sulfides, sulfoxides, but also sulfites and sulfates (sharp peaks indicate inorganic substances or, in general, small molecule compounds) [17], sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), and sulphurous acid (H<sub>2</sub>SO<sub>3</sub>) [19]. They are sourced from detergents, dyes, and catalyst leftovers. As mentioned, the same spectral bands are assigned to sulfuric acid, which is particularly corrosive, contributes to the acidic pH of the water, and, by releasing SO<sub>2</sub>, also contributes to air pollution. Bands at 2606 and 2512cm<sup>-1</sup> are assigned to thiols, sulfides, hydrogen sulfide (H<sub>2</sub>S) [17, 19], a category of pollutants, all releasing sulfur and hydrogen sulfide which is highly toxic, flammable and corrosive.

Organic sulfides (S-) CH<sub>3</sub> and (S-) CH<sub>2</sub> denoted by the 2850, 1453 and 1378cm<sup>-1</sup> bands from plastics (polyphenyl sulfides) [17], that can release gaseous compounds under certain conditions, and landfills are an environment that can promote such decompositions. Among the gaseous compounds that can be released from organic sulfides are hydrogen sulfide (H<sub>2</sub>S) and methane (CH<sub>4</sub>).

Diazo compounds result from textile dyes washed away from the material under rain; humidity conditions are denoted by the 2222 and 2137cm<sup>-1</sup> bands. In the same range of 2222 and 2137cm<sup>-1</sup> bands, together with the bands at 2070 and 2025cm<sup>-1</sup>, are identified organic nitriles [17] and inorganic cyanides (NaCN and KCN at 2070cm<sup>-1</sup>) [17]. The 1322cm<sup>-1</sup> peak is assigned to cyanates and cyanides [17, 19], compounds that present cyanide-like risks when the conditions to release cyan ions are met. Sources may include both household and vegetable waste with a content of cyanohydrins that release HCN, but also plastics such as acrylonitrile, etc. Nitrile compounds are found in many materials that replace natural rubber, including for insulating windows but also for gaskets on various tanks, including cars, and are very resistant to petroleum products (gasoline, oils). Another source is surgical gloves (made of methylcyanoacrylate). Methylcyanoacrylate is also used as an adhesive. They are all potential sources of very toxic CN ions.

The bands at 1631, 1378 and 1322cm<sup>-1</sup> also denote nitrites, nitro, and nitroso organic derivatives, both aliphatic and aromatic [17, 19]. They come from both food dyes and preservatives as well as textile materials. They are harmful insofar as they emit NO. Among nitroderivatives, nitrobenzene is clearly identified by the IR-specific modes at 1547, 1378, 872 and 713cm<sup>-1</sup> [17].

Vibration mods at 3065, 2962 and 2925cm<sup>-1</sup> are assigned to CH-Hal halogenated organic compounds [17, 18] used as foaming agents in sprays and polyurethane foams. Halogenated organic substances are not only potential sources of ignition and explosion but also sources of dioxins and furans. Halide acids, denoted by the bands at 1800 and 600cm<sup>-1</sup> are very corrosive and toxic by releasing halogens (chlorine, fluorine) under certain conditions of sun, moisture, or interactions with other substances.

Methylethers denoted by the 2850cm<sup>-1</sup> band can cause fires and explosions, as can cyclohexanes with vibration modes in the same range, denoted also by the comb at 2755–2606cm<sup>-1</sup> [17]. Cyclohexanes, which are used as solvents and as raw materials for polyamides (Nylon 66), are also very toxic.

#### ***Leachate and emission effects on the surrounding area***

The schematic representation in figure 3a illustrates the constituents identified by rapid tests and spectroscopic analysis in the sample of water collected from a ditch situated in the vicinity of the landfill subject of this study. The possible sources are also indicated in figure 3a.

The effects of the landfill on the surrounding environment are schematically presented in figure 3b.

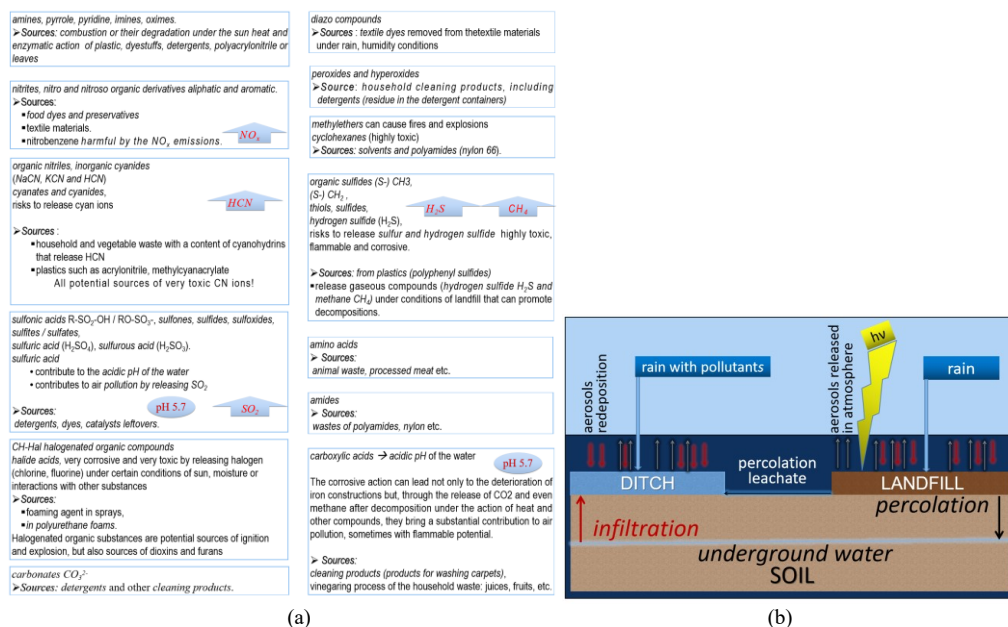


Fig. 3. Pollutants identified in the water of the ditch from the landfill vicinity and their potential sources (a); Effects induced by the landfill in the surrounding environment (b)

The rainwater that percolates through the solid waste in the landfill results in leachate and may also percolate further through the soil into the underground water. Either one way or the other, the pollutants contained in the leachate and/or underground water will infiltrate into the ditch. On the other hand, the emissions in the atmosphere of volatile compounds and particulate matter return to the soil with the rainwater or by "dust" redeposition, and they are also found in the ditch water.

## Conclusions

Even if some of the values obtained from the water analysis do not exceed or are not very worrying—in the sense that they do not seem to be a danger each separately—their cumulative effect can affect health. Also, as a consequence of the cumulative effect of the compounds present in the water in the ditch near the landfill, that water has an acidic character, with consequences not only for vegetation but also for animals—even wild ones—that can consume that water, and their illnesses can even lead to their deaths and the occurrence of additional health hazards for people in the area. Another aspect of the presence of a large number of contaminants is that of the interaction between them, as well as thermal decomposition and under the action of light, which have the effect of releasing flammable and/or potentially explosive compounds, such as methane gas and others.

The quick tests performed as described for the preliminary evaluation of the pollutant chemical species induced in the waters of the ditch situated in the vicinity of the landfill provided qualitative and semi-quantitative information. These kinds of tests may be of high importance in situations when elements of high-risk contamination are suspected or there is a clear indication such as colour, general aspect of the water, or smell in the air. Such rapid and on-site testing can lead to immediate temporary safety measures to prevent the amplification

and spread of contamination, followed later by more elaborate testing to make decisions about the location of the source in order to take specific measures to stop it. The evaluation based on the IR analysis of the vibration modes led to the identification of the types of organic compounds that are found in the water from the ditch in the immediate vicinity of the landfill. The source of contamination was thus confirmed as being the landfill through the various wastes of textiles, food, or cleaning products used in households or in the activities carried out in service workshops specialised in cleaning. Even if the water in that ditch is not used for household or industrial use, its quality reflects the degree of pollution in the environment around the landfill. Those compounds highlighted by the analyses presented reach the groundwater, are released into the air by evaporation, and return to the ground with precipitation or dew. Also worrying is the level of hydrogen cyanide, which has evaporation point temperatures starting at 250°C when part of the HCN remains in the water. The higher the temperature, the greater the release of HCN into the air. If, to the measured values for the release of HCN from the water, is added the contribution of the landfill itself, the image of pollution only with this particularly toxic compound is a worrying one.

Therefore, careful monitoring of areas near landfills in terms of the degree of contamination induced by stored waste is essential to preventing major pollution events. The study shows the importance of quick and low-cost tests for the detection of pollution episodes in the case of closed landfills, for which leachate treatment activities are no longer carried out, as well as other activities to protect the environment against contamination induced by the exposure of garbage to various actions of degradation and emission.

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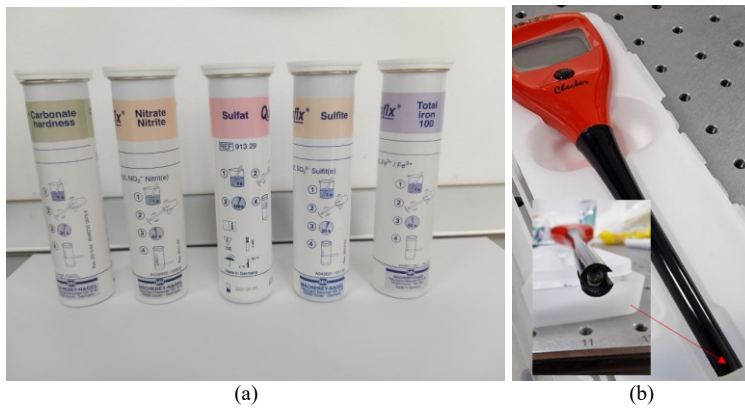


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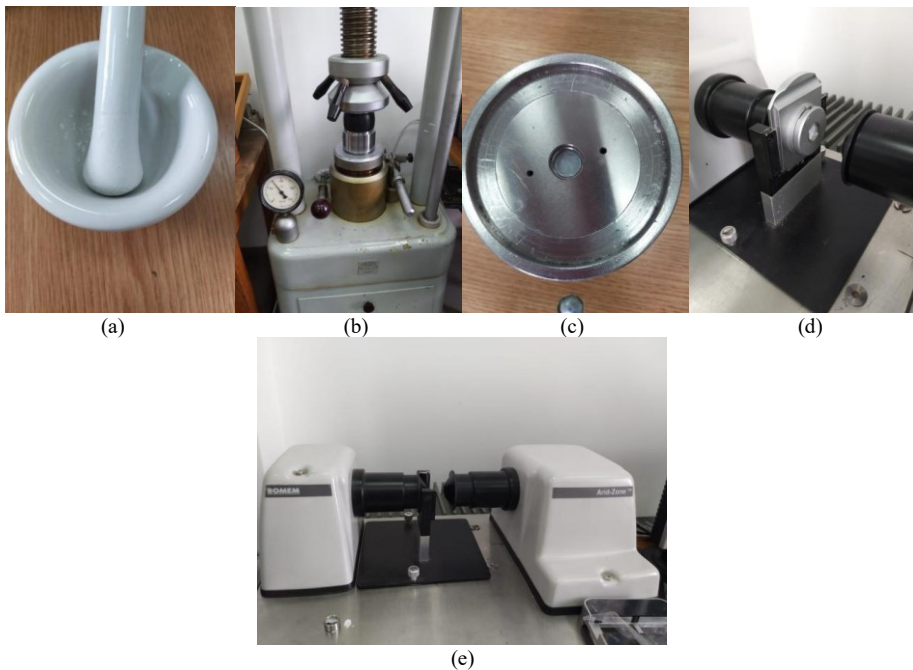
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**Fig. 1 (supplementary).** Quantofix strips containers with the instructions inscription (a) and pH meter with bulb (b)



**Fig. 2 (supplementary).** Images of sample preparation for FTIR analysis: grind mixture of sample incorporated in KBr in the mortar (a), pneumatic press (b), obtained pellet (c), sample holder (d), FTIR Bomem Spectrometer.

**Table 1 (supplementary).** Quantofix strips test instructions. Procedure and interferences.

Tested ions/ Reference number	Dipping time, td (s)	Reading time, tr (s)	Color scale and conversions	Interferences and recommendations for avoiding interferences
CO <sub>3</sub> <sup>2-</sup> ions (carbonate)/ #913 23	5 seconds	30 seconds	0 <sup>0</sup> d, 3 <sup>0</sup> d, 6 <sup>0</sup> d, 10 <sup>0</sup> d, 15 <sup>0</sup> d, 20 <sup>0</sup> d where 1 <sup>0</sup> d =17.8 mg/l CaCO <sub>3</sub>	Interference with strong bases and acid-consuming compounds.
NO <sub>3</sub> <sup>-</sup> ions (nitrate)/ #913 13	1 second	60 seconds	(0, 10, 25, 50, 100, 250, 500) mg/l Color change is read for nitrates in the lower test field on the nitrate/ nitrite strip	Recommended pH range is pH 1 –9 In order to avoid interference with nitrite test, sulfamic acid is recommended to be added.
NO <sub>2</sub> <sup>-</sup> ions (nitrite)/ #913 13	1 second	60 seconds	(0, 1, 5, 10, 20, 40, 80) mg/l Color change is read for nitrites in the upper test field on the nitrate/ nitrite strip	Recommended pH range is pH 1 –9 If pH < 1 the sample must be buffered with sodium acetate. If pH > 9, citric acid is recommended to be add to set to a pH value of 3–5.
SO <sub>4</sub> <sup>2-</sup> ions (sulfate)/ #913 29	1 second	120 seconds	<200, >400, >800, >1200, >16 00 mg/l	Recommended pH to avoid interferences is pH value of 4–8. If pH < 4, the sample must be buffered with sodium acetate. If pH > 8, the sample must be buffered with tartaric acid.
SO <sub>3</sub> <sup>2-</sup> ions (sulfite)/ #913 06	1 second	20 seconds	0, 10, 25, 50, 100, 250, 500, 1000 mg/l	Recommended pH value of 6–9 If pH<6, the sample must be buffered with sodium acetate prior testing. Sulfide ions lead to a red coloration, and when is the case, the interference can be eliminated by adding an excess of nickel (II) ions followed by filtration to eliminate nickel sulfide resulted. Reducing acids such as hydroxylammonium chloride or ascorbic acid can lead to falsely low test results.
Fe <sup>2+/3+</sup> ions (iron)/ #913 44	1 second	60 seconds	(0, 2, 5, 10, 25, 50, 100) mg/l	Interference with coloration produced by heavy metals in higher concentration which can be eliminated with add of cadmium powder. Recommended pH range is pH 1 –7.