BULETINUL INSTITUTULUI POLITEHNIC DIN IAŞI
Publicat de
Universitatea Tehnică „Gheorghe Asachi” din Iaşi
Volumul 71 (75), Numărul 4, 2025
Secţia
CHIMIE şi INGINERIE CHIMICĂ
DOI: 10.5281/zenodo.19660738

DEVELOPMENT OF AN ANALYSIS METHODOLOGY FOR THE CHARACTERIZATION OF LEACHATE SAMPLES

BY

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Received: September 7, 2025
Accepted for publication: December 20, 2025

Abstract. Landfill leachate (leachate) is a liquid generated during the storage process of municipal waste. Due to its complex composition (which often includes highly toxic inorganic and organic compounds), leachate samples must be detailed characterized to ensure their effective treatment and environmental protection. The most commonly used indicators for the characterization of leachate samples are color, temperature, pH, electrical conductivity (1/R), turbidity, total suspended solids (TSS), chemical oxygen demand (COD), ammonium nitrogen ($N\text{-}NH_4^+$), chlorides (Cl^-), and heavy metal content. However, the measured values of these indicators largely depend on the time elapsed between sample collection and their actual determination. In order to obtain accurate values, this study presents a methodology for leachate sample analysis, in which the order of indicator determination was established experimentally. The experimental studies were conducted using the same leachate sample, and each indicator was measured periodically over four days. By following such a methodology, accurate values for the qualitative, physical and chemical indicators characteristic of the leachate samples can be obtained, which may be useful in waste management.

Keywords: leachate sample, characteristic indicators, analytical measurements, experimental methodology.

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1. Introduction

The liquid generated from the decomposition of solid waste, commonly referred to as leachate, represents one of the major issues that must be managed for the sustainable operation of waste landfills, anywhere in the world (Naveen *et al*., 2017; Baettker *et al*., 2020). Generally, landfill leachate is a mixture of rainwater (due to precipitation), water produced by the biological (anaerobic) degradation of waste, and moisture derived from the waste itself (Teng *et al*., 2021; Rhoualem *et al*., 2022). Therefore, any leachate sample, regardless of the landfill from which it is collected, is considered a complex mixture containing large quantities of organic compounds (such as aliphatic hydrocarbons, detergents, pesticides, etc.), simple inorganic salts, and significant amounts of heavy metal ions (Teng *et al*., 2021; Nath and Debnath, 2022). Precisely due to its complex composition, leachate is considered toxic to the environment, and its uncontrolled discharge represents a real danger to ecosystems (Pandey *et al*., 2022; Chen *et al*., 2024).

Numerous technologies, including chemical methods (ex. chemical precipitation, Fenton oxidation, etc.) (Lindamulla *et al*., 2022; Kundu *et al*., 2023), physico-chemical methods (ex. adsorption, membrane processes, etc.) (Castrillón *et al*., 2010; Kumar *et al*., 2025), and biological processes (ex. activated sludge processes) (El-Gohary and Kamel, 2016; Ilmasari *et al*., 2022) have been developed and used for treating leachate resulting from waste deposits. Unfortunately, many of these methods have moderate efficiency or too high implementation costs compared to the total operating costs of landfills (Ilmasari *et al*., 2022; Krause *et al.,* 2023). Therefore, for large-scale applications, a compromise must be found between the performance of the treatment methods and the costs generated by their implementation.

The starting point in finding such a compromise, and therefore in selecting an appropriate treatment method, is the knowledge of the leachate composition as detailed as possible. Consequently, periodic analysis of leachate samples and the determination of physico-chemical indicators are necessary to allow the characterization of these types of samples. Among the indicators used to characterize leachate samples, color, temperature, pH, electrical conductivity (1/R), turbidity, total suspended solids (TSS), chemical oxygen demand (COD), ammonia nitrogen ($N\text{-}NH_4^+$), chlorides (Cl^-) and concentrations of toxic heavy metals (Cr-total, Ni and Pb) are most often imposed by environmental regulations (Ergene *et al*., 2022; Mohammad *et al*., 2022).

However, due to the large number of compounds found in leachate samples, their composition changes continuously, mainly because of secondary processes (such as precipitation, hydrolysis, degradation, or complexation) that occurs (Ilmasari *et al*., 2022). The occurrence of these secondary processes affects the measured values of some of the indicators mentioned above, and can therefore lead to incorrect decisions regarding the selection of leachate treatment methods. Therefore, the determination of these indicators must be carried out in a well-defined order that takes into account the possibility of variation in their values over time, precisely due to these secondary processes.

This study aims to develop an analytical methodology that can be used in the case of leachate samples, taking into account the order and time interval in which certain indicators used to characterize these types of samples can be experimentally determined.

2. Experimental

2.1. Sampling of leachate

The leachate samples used in this study were collected from the area of the Integrated Waste Management Center Moara, located in Vornicenii Mari, Suceava County, Romania (Fig. 1), and were not preserved.

(a) (b) (c)

Fig. 1 – Geographical localization (a), and general aspect (b, c) of the leachate samples used in this study.

The leachate samples were collected in tightly sealed 1-liter plastic bottles, and after sampling, they were transported to the laboratory and kept in the dark at a constant temperature (room temperature). Before being used for laboratory analyses, the leachate samples were filtered (using qualitative filter paper) to remove solid impurities.

2.2. Analytical methods

To characterize the leachate samples, the following indicators were analyzed: color, temperature, pH, electrical conductibility (1/R), turbidity, total suspended solids (TSS), chemical oxygen demand (COD), ammonia nitrogen ($N\text{-}NH_4^+$), chlorides (Cl^-) and some heavy metals (Cr-total, Ni and Pb). The methods used for the analysis of these indicators were those recommended by the standards and are summarized in Table 1. For the determination of each indicator, a leachate sample was collected, brought to the laboratory, and analyzed.

Table 1
The analyzed indicators and the standards used for their determination (MMGA 95/2005)

Indicator	Analytical method	STAS
pH	Potentiometry	SR EN ISO 10523 /2012
Electrical conductibility	Conductometry	ISO 7888:1985
Turbidity	Turbidimetry	SR EN ISO 7027-1:2016
Total suspended solids	Gravimetry	SR EN 872/2009
Chemical oxygen demand	Titrimetry	STAS ISO 15705/2002
Ammonia nitrogen	Spectrophotometry	STAS 8683-70
Chlorides	Titrimetry	ISO 9297/200
Heavy metals (Cr-total,Ni, Pb)	AAS	SR EN ISO 8288:2000

The value obtained was considered the "0" (initial) value of the indicator, and the time interval for all subsequent measurements was measured from that moment. The same leachate sample (stored in the laboratory under normal conditions) was used to perform the experimental measurements for a specific indicator at all time intervals. For the determination of each indicator, the leachate samples were prepared according to the specific methodology described in the standards (Table1).

In each case, measurements were performed in duplicate, and the average values were used in graphical representations.

3. Results and discussion

For the characterization of leachate samples (liquid resulting from the percolation of water through waste), a wide range of physico-chemical indicators are used, regulated at the national level by environmental legislation (MMGA 95/2005). The values determined for these indicators are then used to assess the degree of hazard and to establish appropriate management strategies for the leachate generated in a given landfill. In designing such management strategies, the most accurate possible measurement of each indicator is particularly important, and therefore special attention must be paid to the way experimental measurements are conducted. From an analytical perspective, the indicators used in the characterization of leachate can be divided into three categories: qualitative indicators (color, temperature), physical indicators (pH, electrical conductibility (1/R), turbidity, total suspended solids (TSS)), and chemical indicators (chemical oxygen demand (COD), ammonia nitrogen ($N\text{-}NH_4^+$), chlorides (Cl^-) and some heavy metals (Cr-total, Ni and Pb)) (Wdowczyk and Szymanska-Pulikowska, 2021; Touzani *et al*., 2024). The order in which these indicators are determined is very important for the accuracy of experimental measurements, and therefore an appropriate analysis strategy must be considered.

3.1. Determination of qualitative indicators

From the category of qualitative indicators, color and temperature were analyzed. These measurements are taken on-site, directly in the sampling bottle. All leachate samples collected for this study exhibited a dark brown/black color and a temperature ranging between 12 and 15°C, depending on the month in which the sampling was carried out. The values of the quality indicators do not provide any information regarding the chemical composition of the leachate sample, but their values must be included in the analysis reports, in accordance with legislative requirements.

3.2. Determination of physical indicators

Physical indicators such as pH, electrical conductivity (1/R), turbidity, and total suspended solids (TSS) are commonly used in the characterization of leachate samples. Although none of these indicators provide direct information about the chemical composition, the experimentally measured values allow for determining the acidic or basic nature of the leachate, as well as the concentration of soluble and insoluble salts in the analyzed sample (Abdel-Shafy *et al.*, 2024). Such information's are particularly useful for conducting subsequent chemical analyses. The determination of these physical indicators is carried out exclusively in the laboratory, using experimental methodologies outlined in the standards (Table 1).

To determine the order in which these indicators should be analyzed, their values were measured at different time intervals using the same leachate sample, and the results obtained are illustrated in Fig. 2.

As can be seen in Fig. 2, the values of all physical indicators examined experimentally increase with increasing time since the leachate sample collection. This increase is most likely due to secondary equilibriums (acid-base, redox, precipitation and complexation) occurring within the complex mixture that constitutes the leachate sample, and it further highlights the need to develop a comprehensive analytical strategy.

The highest increase in the measured values is recorded in the case of total suspended solids (TSS) and pH, followed by turbidity and electrical conductibility (1/R), when for each hour that passes since the samples was collected, the experimentally measured values increase by 2.08%, 0.93%, 0.82% and 0.33% respectively. Under these conditions, the time required for the values of these indicators to vary by 5% from their initial value (analytical relevance criterion for experimental measurements) is 1.79 hours for turbidity, 0.29 hours for TSS, 2.59 hours for pH, and 12.7 hours for electrical conductibility (Fig. 2).

This means that after collecting the leachate sample, the experimental measurement of pH, electrical conductivity (1/R), turbidity and total suspended solids (TSS) must be carried out within a maximum of 2 hours to ensure that the measured values meet the analytical accuracy criteria.

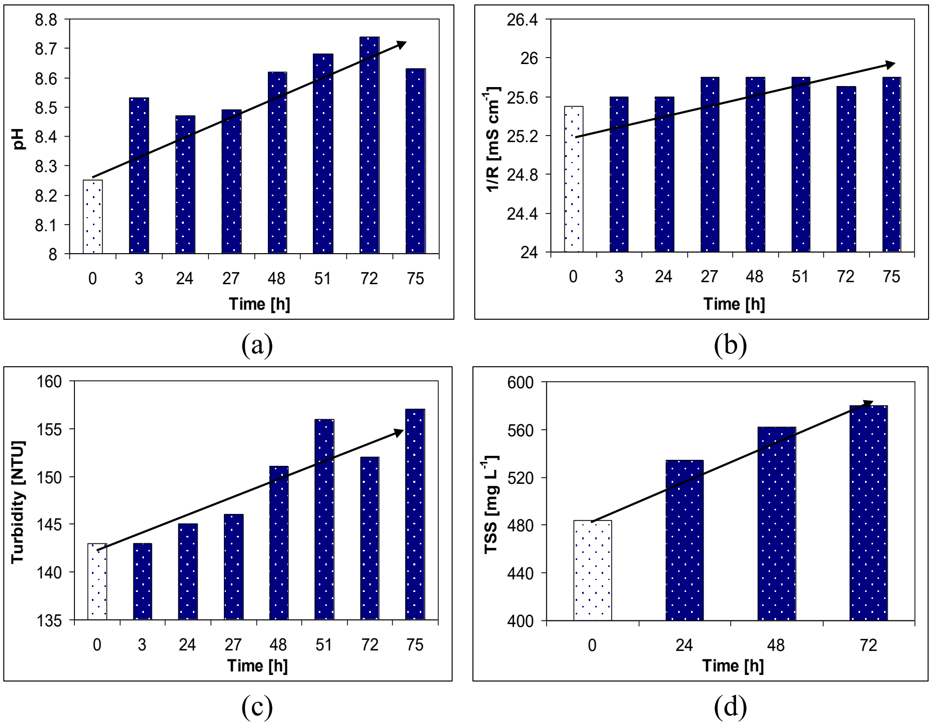

Fig. 2 – Variation of pH (a), electrical conductibility (1/R) (b), turbidity (c), and TSS (d) as a function of time, for the leachate sample collected from the Moara landfill site (Vornicenii Mari, Suceava).

Fortunately, this is possible, because in the case of these indicators, the leachate samples are used directly without any additional preparation, and the analytical methods recommended by the standards (Table 1) are direct, fast and easy to use.

3.3. Determination of chemical indicators

Among the chemical indicators that can be used to characterize leachate samples, chemical oxygen demand (COD), ammonia nitrogen ($N\text{-}NH_4^+$), chlorides (Cl^-) and some heavy metals (Cr-total, Ni and Pb) were selected for this study. This is because the chemical oxygen demand (COD) is a measure of the organic load in the leachate, ammonia nitrogen ($N\text{-}NH_4^+$) is correlated with the degree of waste decomposition, the concentration of chloride ions (Cl^-) provides information about the level of inorganic contamination, and the concentration of heavy metals (Cr-total, Ni and Pb) is most often associated with the toxic potential and contamination risk of the leachate (Anand and Palani, 2022; Qian *et al*., 2024). For each of these chemical indicators, the standards prescribe specific analytical methods that must be followed rigorously (Table 1).

Thus, due to the much more complex experimental methodology, which also involves sample preparation stages for the leachate, the values of the chemical indicators were determined at 24-hour intervals, using the same leachate sample. The results obtained in this case are presented in Fig. 3.

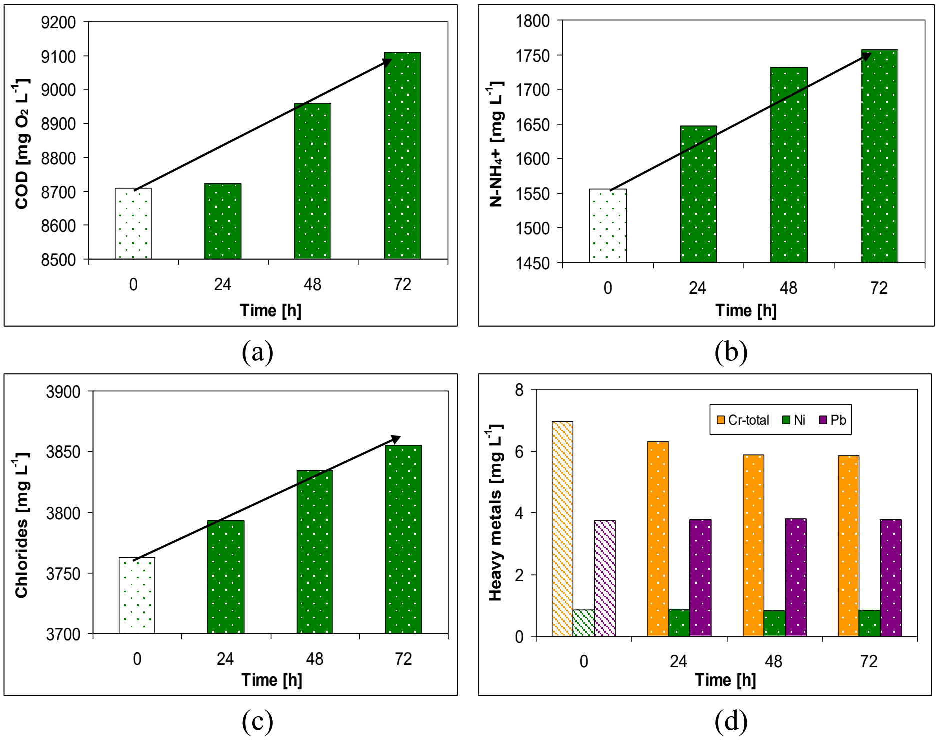

Fig. 3 − Variation of chemical oxygen demand (COD) (a), ammonia nitrogen N-NH_4^+ (b), chlorides (Cl^-) (c), heavy metals (Cr-total, Ni and Pb) (d) as a function of time, for the leachate sample collected from the Moara landfill site (Vornicenii Mari, Suceava).

In the case of chemical indicators chemical oxygen demand (COD), ammonia nitrogen (N-NH_4^+) and chlorides (Cl^-), an increase in the measured values can be observed as a function of time (Fig. 3 (a-c)). These variations indicate that these parameters are also influenced by the secondary equilibriums that continuously occur between the large numbers of components in the leachate sample. For each hour that passes since the leachate sample was collected, the experimentally measured values increase by 0.59% for COD, 3.79% for N-NH_4^+, and 1.25% for Cl^-, respectively. Therefore, the time interval in which the experimentally measured values of these indicators vary by 5% (the threshold for analytical relevance) from their initial value is 4.37 hours in the case of COD, 4.93 hours in the case of $N\text{-}NH_4^+$ and 6.91 hours in the case of Cl^-. This means that the indicators chemical oxygen demand (COD), ammonia nitrogen ($N\text{-}NH_4^+$) and chlorides (Cl^-) can be analyzed after the determination of the physical indicators, as the time required for significant variation in their experimental values is longer. However, even in this case, the analysis should not exceed 6 hours from the moment the leachate sample is collected.

A much smaller variation of the experimentally measured values can be observed for heavy metals (Fig. 3d). After 72 hours, the concentrations of Cr-total, Ni, and Pb varies by only 1.11%, 0.02%, and 0.03%, respectively. This insignificant variation in heavy metal concentrations is due to the fact that, for their determination by atomic absorption spectrometry (AAS) (Table 1), the leachate samples must be mineralized (by treatment with concentrated HNO_3 at 70-80°C). During the mineralization step, the organic compounds in the leachate sample are degraded, and the inorganic compounds are converted into simple forms (free ions). The mineralization step must be continued until a clear, colorless (or slightly yellowish) sample is obtained, suitable for AAS analysis. Such a solution is stable and can be analyzed within a maximum of 5 days. Therefore, once the mineralized sample has been prepared, the determination of heavy metal concentrations in the leachate sample can be left to the end, without the results being affected by errors due to changes in the sample composition.

3.4. Proposed methodology for leachate sample analysis

In general, the characterization of leachate samples involves the experimental measurement of a specific set of indicators (qualitative, physical, and chemical) (Wdowczyk and Szymanska-Pulikowska, 2021). The number of indicators to be analyzed depends on the purpose for which the leachate characterization is required, but at least 2-3 indicators from each category are always selected. This approach provides a more comprehensive understanding of the leachate composition, its toxic and environmental contamination potential, as well as possible valorization strategies.

The results presented in the previous sections have shown that the experimentally measured values for the qualitative, physical or chemical indicators used in the characterization of leachate samples can change over time. This variation in experimental values is mainly determined by the secondary equilibriums that occur in the leachate samples, and is due to the large number of chemical compounds with different reactivity found in their composition. Therefore, in order to determine as accurately as possible the values of each indicator selected for the characterization of leachate samples, an analytical methodology must be designed, which establishes the order in which these indicators should be analyzed.

Such an analysis methodology is presented in Fig. 4, and for its design, the variations over time of the experimental values measured for each analyzed indicator were taken into account.

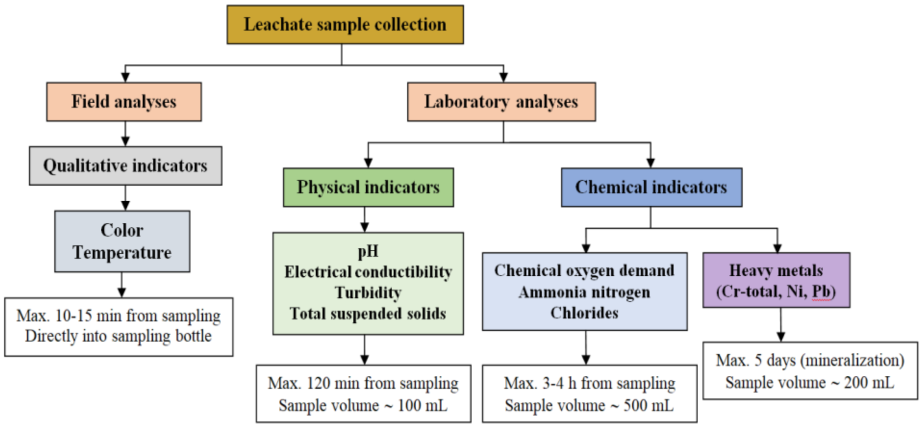

Fig. 4 – Proposed methodology for leachate sample analysis.

The analysis of the leachate sample begins in the field, immediately after sampling, when qualitative indicators (such as color and temperature) are determined directly on site. This avoids possible errors caused by changes in the color of the sample or its cooling/heating. After the field measurements are completed, the leachate sample is transported to the laboratory, where it is used for the determination of quantitative indicators.

The first category of quantitative indicators to be analyzed is that of physical parameters. Thus, the determination of pH, electrical conductibility (1/R), turbidity and total suspended solids (TSS) must be carried out within a maximum of 2 hours (120 minutes) from the leachate sample collection (see Fig. 4). After this time interval, the values of these parameters can vary significantly (see Section 3.2), and this should be avoided. This time interval of 2 hours (120 minute) for determining the physical indicators is sufficient, because a direct method is used for each physical indicator, and the leachate sample does not require any additional preparation steps.

The next step involves the determination of chemical indicators (Fig. 4). A part of the leachate sample (approximately 200 mL) is subjected to the mineralization process and used for the analysis of heavy metals, while another part (approximately 500 mL) is used for the determination of chemical oxygen demand (COD), ammonia nitrogen ($N\text{-}NH_4^+$) and chlorides (Cl^-). Since the mineralization of the leachate sample can take several hours, it is recommended that this process be carried out simultaneously with the determination of the other chemical indicators. Once the leachate sample has been mineralized and transferred into a volumetric flask, the analysis of heavy metal content can be done in a maximum of 5 days with high accuracy.Simultaneously with the mineralization of the sample, analyses for determining chemical oxygen demand (COD), ammonia nitrogen ($N\text{-}NH_4^+$) and chlorides (Cl^-) should be started. The methods used for determining each of these indicators are more complex (Table 1), and therefore the organization of work time is essential. Considering the way the values of these indicators vary over time, chemical oxygen demand (COD) should be analyzed first, followed by ammonia nitrogen ($N\text{-}NH_4^+$) and then chlorides (Cl^-) (see Section 3.3). However, the maximum time in which these indicators should be analyzed must not exceed 4 hours, and the required sample volume is approximately 500 mL.

By following such a methodology, accurate values for the qualitative, physical and chemical indicators characteristic of the leachate samples can be obtained, which may be useful in waste management.

4. Conclusions

This study proposes a methodology for the analysis of leachate samples, which can be applied at laboratory scale to determine their characteristic indicators. To design this methodology, qualitative indicators (color, temperature), physical indicators (pH, electrical conductibility (1/R), turbidity and total suspended solids (TSS)) and chemical indicators (chemical oxygen demand (COD), ammonia nitrogen ($N\text{-}NH_4^+$) and chlorides (Cl^-)) were selected. All these indicators were determined at different time intervals using the same leachate sample. The experimental results showed that the measured values for each indicator vary over time. The experimental results showed that the measured values for each indicator vary over time. Therefore, it is recommended that qualitative indicators be determined immediately after leachate sampling (on-site), physical indicators within a maximum of 2 hours after sampling, and chemical indicators within a maximum of 4 hours. Among the chemical indicators, heavy metals are an exception, as their concentration can be determined within up to 5 days after the mineralization of the leachate sample. In this way, accurate values of the qualitative, physical, and chemical indicators characteristic of leachate samples are obtained, which can be used in waste management.

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ELABORAREA UNEI METODOLOGII DE ANALIZĂ PENTRU CARACTERIZAREA PROBELOR DE LEVIGAT

(Rezumat)

Levigatul din depozitele de deşeuri este un lichid generat în timpul procesului de depozitare a deşeurilor municipale. Datorită compoziţiei sale complexe (care include adesea compuşi anorganici şi organici extrem de toxici), probele de levigat trebuie caracterizate detaliat pentru a asigura tratarea eficientă a acestora. Cei mai utilizaţi indicatori pentru caracterizarea probelor de levigat sunt culoarea, temperatura, pH-ul, conductivitatea electrică (1/R), turbiditatea, solidele totale în suspensie (TSS), consumul chimic de oxigen (COD), azotul amoniacal ($N\text{-}NH_4^+$), clorurile (Cl^-) și conținutul de metale grele. Din păcate, valorile măsurate ale acestor indicatori depind în mare măsură de timpul scurs între colectarea probelor de levigat și determinarea lor efectivă. Pentru a obține valori precise, acest studiu prezintă o metodologie pentru analiza probelor de levigat, în care ordinea de determinare a indicatorilor a fost stabilită experimental. Studiile experimentale au fost efectuate utilizând aceeași probă de levigat, iar fiecare indicator a fost măsurat periodic pe parcursul a patru zile. Respectând o astfel de metodologie, se pot obține valori precise pentru indicatorii calitativi, fizici și chimici caracteristici probelor de levigat, ceea ce poate fi util în strategiile de gestionare a deșeurilor.