



## ANALYSIS OF LEACHATE FROM A NON-SANITARY LANDFILL AND ASSESSMENT OF REMOVAL EFFICIENCY OF *CAFFEINE* USING MATERIAL AND SUBSTANCE FLOW ANALYSIS

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**Abstract:** Leachate from a non-sanitary municipal solid waste landfill (L) was collected to perform physico-chemical (temperature, pH value, conductivity, COD, BOD<sub>5</sub>, NH<sub>4</sub><sup>+</sup>-N, SO<sub>4</sub><sup>2-</sup>, P<sub>tot</sub>, orthophosphates, turbidity, suspended solids) and microbiological (number of aerobic heterotrophs, total number of coliform bacteria, total number of faecal coliform bacteria, number of intestinal enterococci) analyses. Obtained results indicate that landfill L was in methanogenic phase of waste decomposition and included the presence of two species of microorganisms: *Escherichia coli* and *Klebsiella pneumonia*. Additionally, a semi-quantitative screening analysis of collected samples was performed using QP2010-Ultra GC-MS. Sixteen organic compounds were identified, of which the following five belonged to the group of pharmaceuticals: *caffeine*, *22-tricosenoic acid*, *4-methylthiomorpholine-1,1-dioxide*, *2,6,10,14-hexadecatetraen-1-ol,3,7,11,15-tetramethyl-,acetate,(E,E,E)-*, and *1,2-bis(trimethylsilyl)benzene*. The software package STAN was used to conduct material and substance flow analysis. Scenario modeling was performed to assess the removal efficiency of selected technologies in terms of reducing the concentration of *caffeine* because of its well-known negative effects on humans and the environment. Technologies for leachate treatment, which were observed within the Scenario 1, included the hybrid wetland system, membrane bioreactor (MBR) and reverse osmosis (RO). The modeling results indicate the high removal efficiency of caffeine concentration (99.99%).

**Keywords:** landfill leachate; pharmaceuticals; caffeine; STAN



## ANALIZA PROCJEDNE VODE NESANITARNE DEPONIJE I PROCJENA UČINKOVITOSTI REDUKCIJE KOFEINA PRIMJENOM ANALIZE TOKOVA MATERIJALA I SUPSTANCI

**Sažetak:** U sklopu istraživanja analizirana je procjedna voda iz obodnog kanala nesani tarne deponije čvrstog komunalnog otpada unutar lokaliteta L. U prikupljenim uzorcima vode određivani su: osnovni fizikalno-kemijski (temperatura, pH vrijednost, električna vodljivost, HPK, BPK<sub>5</sub>, NH<sub>4</sub><sup>+</sup>-N, SO<sub>4</sub><sup>2-</sup>, ukupni fosfor, ortofosfati, zamućenost i suspendirane tvari) i mikrobiološki parametri (broj aerobnih heterotrofa, ukupan broj koliformnih bakterija, ukupan broj koliformnih bakterija fekalnog porijekla, broj crijevnih enterokoka). Analizom rezultata osnovnih fizikalno-kemijskih parametara, može se zaključiti da se deponija nalazi u metanogenoj fazi degradacije otpada. Rezultati mikrobiološke analize ukazuju na prisutnost dvije vrste mikroorganizama: *Escherichia coli* i *Klebsiella pneumonia*. Primjenom QP2010-Ultra GC-MS realizirana je i polukvantitativna probirna analiza prikupljenih uzoraka, kojom je utvrđena prisutnost 16 organskih spojeva, od kojih 5 pripada skupini farmaceutika: kofein, 22-trikozanoična kiselina, 4-metiltiomorfolin-1,1-dioksid, 2,6,10,14-heksadekatetraen-1-ol, 3,7,11,15-tetrametil-, acetat, (E,E,E)-, 1,2 bis(trimetilsilil)benzen. Primjenom softverskog paketa STAN provedena je analiza tokova materijala i supstanci i realizirano je modeliranje scenarija radi procjene učinkovitosti selektiranih tehnologija za tretman procjednih voda s gledišta redukcije koncentracije stimulansa kofeina. Tehnologije za tretman procjednih voda koje su razmatrane u sklopu Scenarija 1 su hibridni sustav mokrih polja, membranski bioreaktor (MBR) i reverzna osmoza (RO). Rezultati modeliranja pokazali su visoku učinkovitost redukcije koncentracije kofeina, u vrijednosti od 99.99 %.

**Ključne riječi:** procjedne vode deponije; farmaceutici; kofein; STAN



## 1 INTRODUCTION

Leachate is generated through complex chemical reactions, infiltration of atmospheric water in landfill body, water contained in waste, and dissolution of pollutants from waste. Filtrate water of atmospheric origin, constitutive water, as well as water generated in the body of a landfill form the liquid in which polluting matter of organic and inorganic origins is released. This has an adverse impact on the quality of landfill leachate [1, 2]. Because of its toxic composition, leachate pollutes soil and groundwater.

Predicting the actual composition of landfill leachate is difficult because of the complex dynamics of ongoing processes in the landfill body and numerous other variable factors. Registered and identified hazardous and toxic matter are aromatic hydrocarbons, halogenated components, phenols, pesticides, heavy metals, and nutrients [1]. The latest research indicates the importance of identifying and quantifying of pharmaceuticals broadly used in human and veterinary medicine as well as in the form of food supplements. Intake of pharmaceuticals environmentally is possible through several means: inadequate disposal of pharmaceutical products, use of supplements and medication, inefficient treatment of utility and industrial wastewater, and through accumulation within activated sludge and inadequate treatment of the output flow [3].

The main objective of this study was to determine the quality of leachate collected at a non-sanitary municipal solid waste landfill (*L*) in the region of Vojvodina, Republic of Serbia. Due to potentially adverse effects on the human body and environment, the emphasis in this work was on identifying the possibility of reducing the concentration of pharmaceutical *caffeine* using material and substance flow analysis.

## 2 MATERIALS AND METHODS

### 2.1 Collecting of leachate samples from landfill site *L*

Leachate samples were collected during the winter of 2019 within a time interval of 2 h from the peripheral canal of a non-sanitary landfill site *L* (Figure 1). During the process of sampling, 4.5 l of leachate were collected: 2 l, 2 l, and 0.5 l for screening, physico-chemical, and microbiological analyses, respectively. The samples were delivered to the laboratory and stored at a temperature of 4 °C until the moment of preparing the samples for the analysis. The analysis of chemical parameters and the screening analysis were carried out at the accredited Laboratory for monitoring landfills, wastewater and air of the Department of Environmental Engineering and Occupational Safety of the Faculty of Technical Sciences in Novi Sad. Microbiological parameters were determined at the Institute of Public Health of Vojvodina in Novi Sad.



Figure 1 Peripheral canal of the landfill site *L* and process of collecting leachate samples



## 2.2 Analysis of leachate at the landfill site L

Within physical and chemical analyses of collected samples of leachate, field measurements and laboratory analysis of the following parameters were carried out: temperature of leachate, pH value, electrical conductivity, COD, BOD<sub>5</sub>, NH<sub>4</sub><sup>+</sup>-N, SO<sub>4</sub><sup>2-</sup>, total phosphorus, orthophosphates, turbidity, suspended matter. The parameters pH value and electrical conductivity were determined using a multiparameter device (WTW Multi 340i) in the field. Laboratory analyses of non-organic parameters was conducted using BOD Trak HACH and UV-VIS spectrophotometer HACH DR5000 devices. The following methods were applied in the physical and chemical analyses: EPA 150.1, EPA 120.1, EPA 365.3, EPA 160.2, HACH LCK 303, HACH LCK 114, HACH Method 8051, and HACH BOD TRAK [4].

For the screening analysis, the sample was prepared using liquid-liquid extraction and concentrated using a Kuderna-Danish device. The previously prepared internal standard Fenantren D10 was applied for the liquid-liquid extraction, with a concentration of 15 ppm in methanol, while dichloromethane was used as a solvent. Shimadzu QP2010-Ultra and Agilent HP-5ms column (30 m × 0.25 mm × 0.25 μm) was used in the analysis.

Within the microbiological analysis the identification of four parameters were carried out, namely the number of aerobic heterotrophs, the total number of coliform bacteria, the total number of coliform bacteria of faecal origin, detection and identification of the number of intestinal enterococci. These parameters were determined using the following methods: Q3.BP.618, Q3.BP.614, and Q3.BP.615 [5-8].

## 2.3 Modeling and assessment of the efficiency of caffeine reduction using material and substance flow analysis

The software *STAN* is a new user-friendly freeware that supports performing material flow analysis (MFA) according to the Austrian standard ÖNORM S 2096 (MFA–Application in waste management) under consideration of data uncertainties [9].

After a graphical model with predefined components (flows, processes, subsystems, system boundaries, and text fields) is constructed, known data (mass flows, stocks, concentrations, and transfer coefficients) with corresponding physical units can be either entered or imported for different hierarchical layers (good, substance, energy) and time periods. A graph of the model with flows displayed as Sankey arrows (i.e., where the width of a flow is proportional to its value) can be printed or exported into various graphical formats. If sufficient data are given, calculation algorithms can be used to reconcile uncertain data and compute unknown variables, including their uncertainties (error propagation). Statistical tests are used to detect gross errors in a given dataset [9].

Through use of the software package *STAN* (version 2.5.1302), material and substance flow analysis were conducted in this study. Specifically, Scenario 1 was modeled for the purpose of assessing the efficiency of selected technologies for leachate treatment in terms of reducing the concentration of *caffeine* as a stimulus. Technologies for treatment of leachate considered within Scenario 1 included a hybrid system of wetlands, a membrane bioreactor (MBR) and reverse osmosis (RO).

When daily and annual volumes of generated leachate within Scenario 1 were collected, the following formula was applied [10]:

$$Q_f = k(P + Q)/365 \quad (1)$$

where:

Q<sub>f</sub> = daily volume of filtrate [m<sup>3</sup> day<sup>-1</sup>];

k = coefficient characterized by ability to absorb moisture and evaporation of waste;

P = total annual volume of precipitation on the surface of waste [m<sup>3</sup> year<sup>-1</sup>];

Q = total annual volume of water distributed on the surface of waste [m<sup>3</sup> year<sup>-1</sup>];

F = surface [ha].

Data applied when calculating daily and annual volumes of generated leachate were derived from real sources. Data referring to the surface of a hypothetical landfill site was projected based on real data of the exploited surface of landfill L, and amounted to 88,000 m<sup>2</sup>. The annual volume of precipitation was measured on the level of the nearest synoptic station in the period of 1981–2010, and was 614.2 mm year<sup>-1</sup> [11, 12].

When the methodology is applied, it is necessary to consider the fact that 70% of precipitation evaporates from the landfill through drying, and 25% evaporates through biometric processes occurring in the layer of deposited



waste at a depth of 0.5–2 m. Remaining precipitation, as a filtrate, migrates down to the bottom of the landfill by 5–15% for a sloped area and by 1–10% for the area that is not sloped. Consequently, the coefficient  $k$  for landfill sites on a flat terrain is 0.1, whereas for sloped landfill sites (mostly 2%), it is 0.15 [10]. Furthermore, the total annual volume of water distributed on the surface of waste actually accounts for merely 30% of the total annual precipitation remaining after evaporation off the landfill site surface.

In calculations, the following formula is used for converting values from the level of  $\text{mm year}^{-1}$  to  $\text{m}^3 \text{ year}^{-1}$  [13]:

$$\text{mm year}^{-1} = \frac{10^{-3} \text{m}^3}{1 \text{m}^2} \text{ year}^{-1} \quad (2)$$

When (2) was applied, it could be concluded that the annual volume of precipitation per  $1 \text{m}^2$  was  $0.6142 \text{m}^3$ . With the exploited area of landfill ( $L$ ) introduced, which was  $88\,000 \text{m}^2$ , the annual volume of precipitation for the given surface was calculated at  $54\,049.6 \text{m}^3 \text{ year}^{-1}$ . Therefore, a factor  $P$  was determined within (1). Based on the obtained results, the factor  $Q$  was calculated and represented the remaining 30% of the total annual precipitation distributed on the surface of waste, which amounted to  $16\,214.88 \text{m}^3 \text{ year}^{-1}$ . Based on the presented data, an assessment of generating leachate was conducted within the hypothetical landfill site on a daily (3) and annual (4) basis.

$$Q_f = 0.10 \cdot (54\,049.6 \text{m}^3 \text{ year}^{-1} + 16\,214.88 \text{m}^3 \text{ year}^{-1}) / 365 = 19.25 \text{m}^3 \text{ day}^{-1} \quad (3)$$

$$Q_f = 0.10 \cdot (54\,049.6 \text{m}^3 \text{ year}^{-1} + 16\,214.88 \text{m}^3 \text{ year}^{-1}) = 7\,026.45 \text{m}^3 \text{ year}^{-1} \quad (4)$$

Concentration of the pharmaceutical *caffeine*, applied as an input concentration within the Scenario 1, was determined through semi-quantification during the screening analysis of the leachate samples from landfill site  $L$ . The concentration of *caffeine* was determined to be  $57.84 \text{mg l}^{-1}$ . Based on the values presented in the literature, the value of efficiency for reducing the flow of *caffeine* using the hybrid system of wetlands through primary use of the plant species *Typha angustifolia* was set to 88.4% [14,15]. The values listed in the literature for the efficiency of reducing the flow of *caffeine* using an MBR was 95%, whereas for reverse osmosis (RO), it was 20% [16-19].

### 3 RESULTS AND DISCUSSION

Results of physical and chemical analyses of leachate from the peripheral canal of landfill  $L$  are presented in Table 1.

Values obtained for the organic load parameters COD and  $\text{BOD}_5$ , as well as their relation  $\text{BOD}_5/\text{COD}$ , indicate the age of the site, historical waste disposal on the selected site, and also that the landfill was in a methanogenic phase. By comparing the results of physical and chemical analyses presented in Table 1 and the results of earlier research, leachate from landfill site  $L$  could be categorized as old leachate [2].

**Table 1 Values of physical and chemical parameters of leachate from landfill site  $L$**

Parameter	Unit	L
Leachate temperature	[°C]	5.2
pH	[-]	7.68
Electrical conductivity	[ $\mu\text{S cm}^{-1}$ ]	1 130
COD	[ $\text{mg l}^{-1}$ ]	956
$\text{BOD}_5$	[ $\text{mg l}^{-1}$ ]	220
$\text{BOD}_5/\text{COD}$	[ $\text{mg l}^{-1}$ ]	0.23
Ammonium ion	[ $\text{mg l}^{-1}$ ]	56
Sulphates	[ $\text{mg l}^{-1}$ ]	46.4
Total phosphorus	[ $\text{mg l}^{-1}$ ]	1.168
Orthophosphates	[ $\text{mg l}^{-1}$ ]	0.378
Turbidity	[NTU]	11.2
Suspended matter	[ $\text{mg l}^{-1}$ ]	7.4



Results of the microbiological analysis of leachate from the peripheral canal of landfill *L* are presented in Table 2. Based on the results of microbiological analyses presented in Table 2, the sample corresponded to the limit values defined in the *Decree on limit values of the emission of pollutants in water and time limits for their reaching* ("Official Gazette of the RS", no. 67/11, 48/12 and 01/16). Bacteria *Escherichia coli* (37 °C, 44 °C) and *Klebsiella pneumoniae* (37 °C, 44 °C) were detected in the tested leachate sample. The screening analysis of the collected leachate sample from the landfill site *L* revealed the presence of 16 organic compounds, of which five fell into a group of pharmaceuticals. Detected pharmaceuticals are listed in Table 3.

One pharmaceutical detected in the leachate of the landfill site *L*, namely, *caffeine*, has been referenced in *NORMAN* (Network of reference laboratories, research centres and related organisations for monitoring of emerging environmental substance) list of emerging substance since 2013.

**Table 2 Values of microbiological parameters of leachate from the landfill site *L***

Parameter	Unit	L	Limit value <sup>1</sup>
Number of aerobic heterotrophs	cfu mL <sup>-1</sup>	10 182	-
Total number of coliform bacteria	cfu 100 mL <sup>-1</sup>	1 100	10 000
Total number of coliform bacteria of faecal origin	cfu 100 mL <sup>-1</sup>	500	2 000
Number of intestinal enterococcus	cfu 100 mL <sup>-1</sup>	100	400

<sup>1</sup> *Decree on limit values of the emission of pollutants in water and time limits for their reaching* ("Official Gazette of the RS," no. 67/11, 48/12 and 01/16), Article 13, Table 4; Annex 2, Chapter III Utility waste water

**Table 3 Pharmaceuticals detected in leachate from the landfill site *L***

Compound	CAS Number	Molecular Formula	Molecular Weight [g mol <sup>-1</sup> ]	Retention Time [min]
caffeine	58-08-2	C <sub>8</sub> H <sub>10</sub> N <sub>4</sub> O <sub>2</sub>	194.19	73.978
22-tricosenoic acid	65119-95-1	C <sub>23</sub> H <sub>44</sub> O <sub>2</sub>	352.60	96.058
4-methylthiomorpholine-1,1-dioxide	25343-91-3	C <sub>5</sub> H <sub>11</sub> NO <sub>2</sub> S	149.21	106.218
2,6,10,14-hexadecatetraen-1-ol, 3,7,11,15-tetramethyl-, acetate, (E,E,E)-	61691-98-3	C <sub>22</sub> H <sub>36</sub> O <sub>2</sub>	332.52	116.618
1,2-bis(trimethylsilyl)benzene	17151-09-6	C <sub>12</sub> H <sub>22</sub> Si <sub>2</sub>	222.48	125.488

The MFA is a method applied with the goal of designing and providing information on anthropogenic systems (anthropogenic metabolism). This method represents a systemic means of assessing flows and stocks of matter within the system defined in space and time, whereby the sources, paths, and indirect and final disposition of matter are connected [20]. The analysis of material flows is a tool for interpreting metabolic processes over the course of time developed complex anthropogenic systems for the purpose of optimization by supporting the making of decisions related to a design of new systems [21]. It is possible to implement mentioned method on two levels, the level of goods and the level of substances, whereby it is called the substance flow analysis (SFA). It is possible to identify the most significant processes during the life cycle of the matter, as well as the detection of relevant matter stocks in the atmosphere and environment, the determining of losses emitted in the environment and the final destinations by interpreting the results of the material flow analysis [20].

Through MFA and SFA using *STAN* software version 2.5.1302, the modeling of Scenario 1 was implemented for a utility solid waste leachate treatment plant using the technologies of hybrid wetlands, MBR and RO. Additionally, their efficiencies in terms of reducing *caffeine* concentration were assessed. The different flows of *caffeine* for Scenario 1, MBR and the hybrid system of wetlands are depicted in Figures 2–4, respectively.

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The results of modeling indicated a high efficiency of 99.99% for reducing the concentration of *caffeine* with Scenario 1.

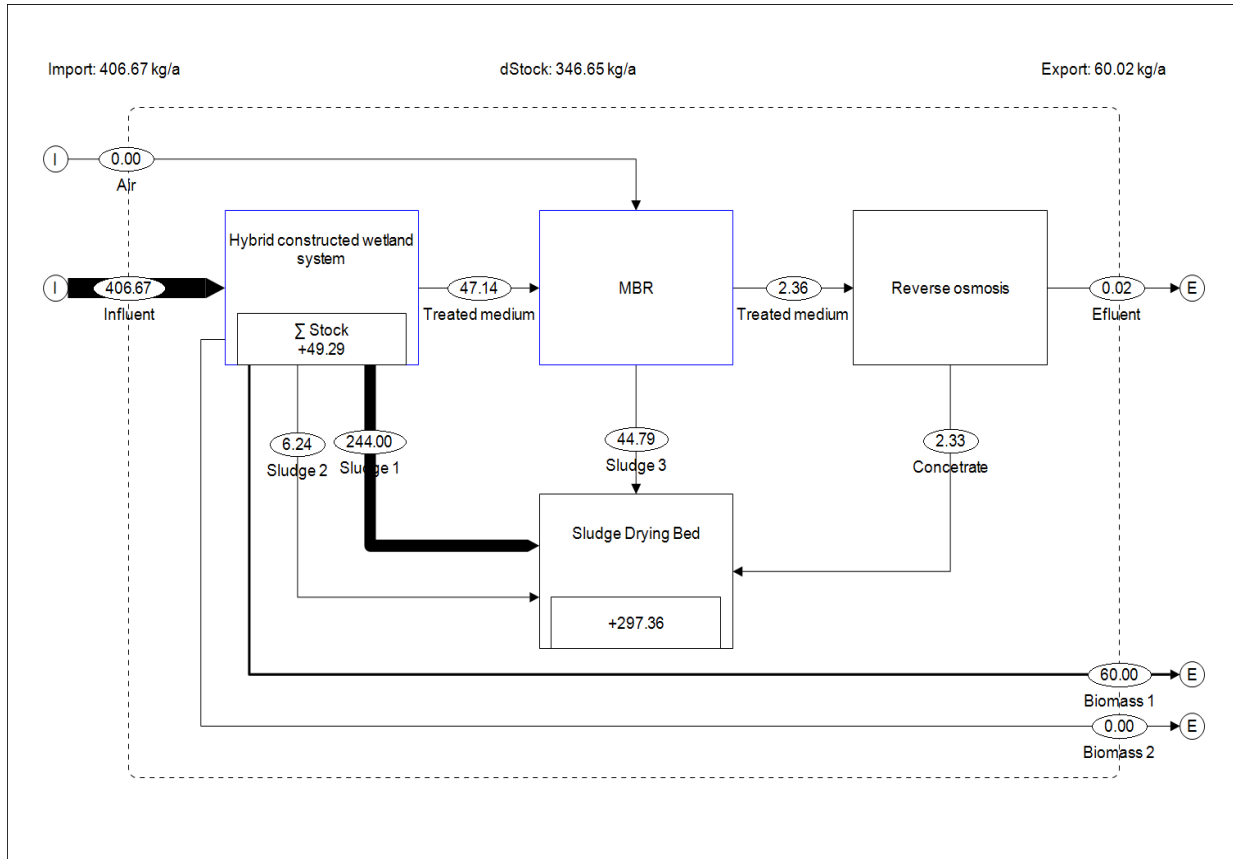


Figure 2 Flow of *caffeine* within Scenario 1

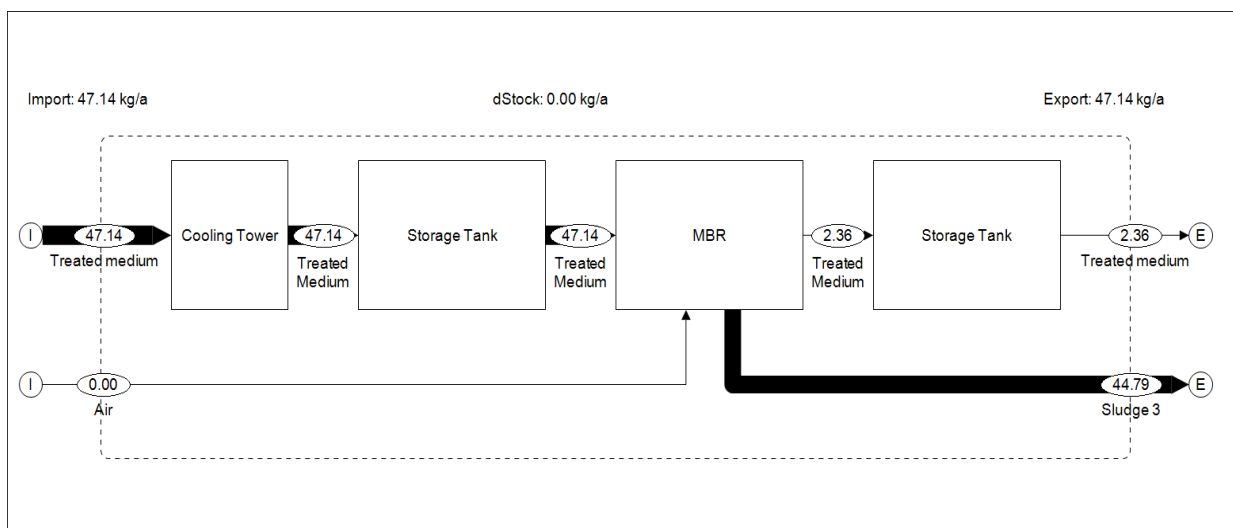


Figure 3 Flow of *caffeine* within the membrane bioreactor (MBR)

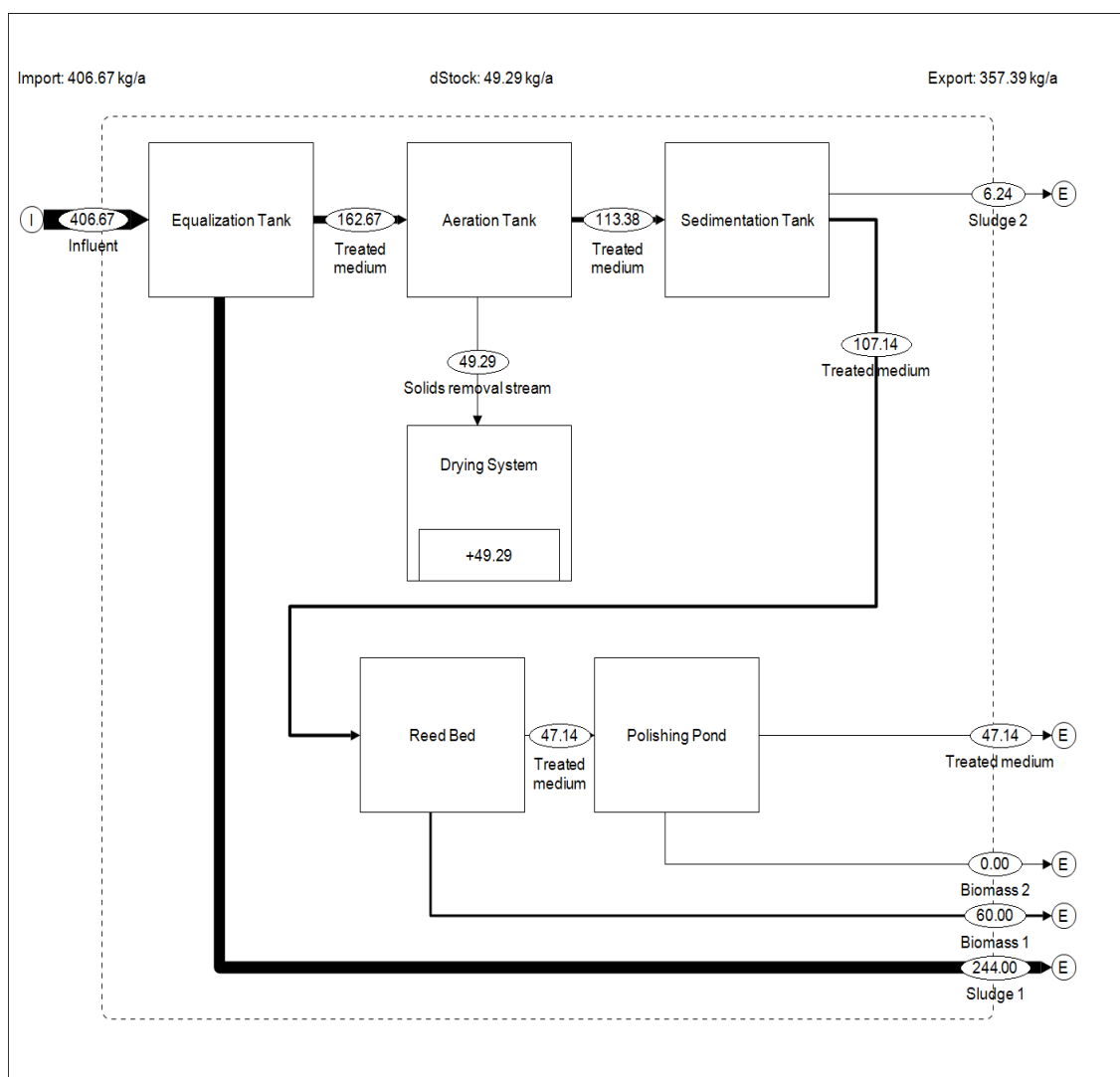


Figure 4 Flow of *caffeine* within the hybrid system of wetlands

#### 4 CONCLUSION

Research results from this study indicated poor quality of leachate from the landfill site *L*, which may have been the result of historically inadequate disposal of various types of waste. Based on a microbiological examination, the leachate sample from the landfill site *L* corresponded to limit values stipulated by national regulations. The specific hazards to human health and the environment are the presence of pharmaceuticals, such as caffeine, 22-tricosenoic acid, 4-methylthiomorpholine-1,1-dioxide, 2,6,10,14-hexadecatetraen-1-ol, 3,7,11,15-tetramethyl-, acetate, (E,E,E)-, and 1,2-bis(trimethylsilyl)benzene. These substances must be included in the databases of future scientific and research programs that monitor leachate, and selected substances and their eco-toxicities must be quantified. Additionally, further optimizing the efficiencies of conventional and alternative treatments of municipal solid waste landfill leachate in terms of reducing concentration of pharmaceuticals is necessary.

In this study, through the use of the software package *STAN*, MFA and SFA were conducted. Specifically, Scenario 1 was modeled for the purpose of assessing the efficiencies of selected technologies for leachate treatment in terms of reducing concentration of *caffeine* as a stimulus. The results of modeling indicated high efficiency (99.99%) for reducing the concentration of *caffeine* with Scenario 1.





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