

Safety evaluation of the leaching of metals from the printed graphic product wastes

ABSTRACT

Due to the technological development of the graphic production, the environment is being faced with a large amount of printed graphic product wastes, especially packaging materials (paper, cardboard, paper and plastic bags, films, etc), but it is also being faced with the problem of their disposal. Many printing inks and coatings used in the production of the printed graphic product contain metals which, after the disposal of graphic waste, can migrate to different systems and have a negative influence on the environment. Because of that, the concentration levels of metals (zinc, copper, chromium, cadmium, lead, and nickel) in the printed graphic product wastes have firstly been determined, and then the impact of those metals, through their migration from the printed graphic product wastes to the simulated environmental mediums with different pH values (acidic and neutral), has been estimated. Based on the experimentally obtained concentrations of metals that have migrated from the printed graphic product wastes to the neutral solution and based on the theoretical distribution coefficient, the concentration of metals in the soil of illegal and municipal landfills, which represents the contribution to the overall metal concentration in the soil due to the migration from the waste printed graphic materials, has been calculated. Also, a comparison between the experimentally obtained metal concentrations and the literature values has been conducted, and an evaluation of their influence on the quality of soil has been given.

KEY WORDS

printed graphic product, waste, metals, leaching tests, landfill

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Introduction

There are data on the generation of municipal waste, degree of recycling, i.e. waste usage, the assessment of the influence of waste on the environment, etc (Prechthai et al., 2008; Vujić et al., 2010). However, there are no data on the contribution of specific components of municipal waste (metals, organic pollutants, etc) to the share of the overall migration to individual environmental mediums. Waste materials being generated as the products of printing industry represent a significant part in the overall quantity of the generated municipal waste. Materials being used in the printing industry are very diverse and complex and they influence the quality of graphic products to a great extent. The structure and characteristics of the materials and their exploitative properties determine the optimal printing technique.

The environment is, due to the technological development of the printing production, being faced with a large amount of printed graphic product waste (paper and plastic bags, cans, old paper, cellophane, posters, cardboard, etc), and later with the problem of their disposal. The printed graphic product wastes may contain various metals potentially dangerous to the environment that come from dyes and pigments, fillers, stabilizers, varnishes, adhesives, etc. Metals as hazardous pollutants can migrate after the disposal to different mediums and have a negative influence on the environment, primarily on the soil of illegal and municipal landfills, but also on the leachates wastewater (Kasassi et al., 2008; Long et al., 2011). Most of the printing ink pigments used to be based on the metallic compounds such as zinc, copper, lead, and chromium. However, harmful metals such as lead and chromium have been prohibited by law in most

of the countries from being used in food packaging (Kim et al., 2008). Heavy metals, such as chromium, cadmium, copper, zinc, and nickel, strongly affect human health (Nouri et al., 2010). Because of their persistence and toxicity, these species can have a serious impact if released into the environment as a result of bioaccumulation, and they may be extremely toxic even in trace quantities (Mahvi et al., 2010). In this regard, the prevention of environmental pollution has actively been studied.

Most of the packaging is poorly designed so that the inner and the outer layers do not maintain structural integrity, thus enabling the components of the printing inks in the outer layer of the packaging and in the very material to migrate to the alimentary product (Kim et al., 2008), but to what insufficient amount of attention has been paid to is the migration of those same components to the environment (Long et al., 2011). Aside from the printing inks, the origin of metals could also belong to specific phases of the technological printing process and the materials used during printing (Adamović et al., 2013; Monte et al., 2009).

In the first phase of this study, the concentrations of metals (zinc, copper, chromium, cadmium, lead, and nickel) in the printed graphic product wastes have been determined, and then in the second phase the influence of their migration from the tested materials to certain simulated environmental mediums has been estimated via leaching (migration) tests. Also, the assessment of the contribution of the concentrations of zinc, nickel and copper in the soil of illegal and municipal landfills has been performed based on the theoretical distribution coefficient, K_{sw} and the concentration of metals that have migrated from the printed graphic product waste to the neutral solution.

Materials and Methods

In this paper, we used the following printed graphic product wastes: a toilet paper package (T)- printed on polyethylene film (PE), a cookie package 1 (C1)- printed on pearlescent polypropylene film (SPP), a cookie package 2 (C2)- printed on oriented polypropylene film (OPP), a candy package (CA)- printed on pearlescent polypropylene film (SPP), a coffee package (C)- printed on metalized polypropylene foil (MPP), a daily newspaper (DN)- printed on roto paper (RP), and a poster (P) - printed on coated paper (CP). The tested samples of polymer packaging materials were printed with CMYK (cyan, magenta, yellow and black) processing flexo inks, which are multi-component mixtures of liquid pigment concentrates based on polyurethane binders and organic solvents (TORDA-ZVEZDA manufacturer, Serbia), whereas the daily newspaper was printed with CMYK processing web-fed offset printing inks and the

poster with the CMYK processing sheet-fed offset printing inks (TOYO INK manufacturer CO., LTD., Japan).

The concentration levels of metals (zinc, copper, chromium, cadmium, lead, and nickel) in printed graphic product wastes were determined according to the following procedure (Kim et al., 2008): 10 g of printed graphic product waste samples were weighed in the ceramic crucible.

After the addition of 5 mL of $Mg(NO_3)_2$ in ethanol (96%), the samples were heated on the hot plate until the cessation of the separation of yellow nitrogen oxide fumes, and then fired in a furnace at 450 °C for 4 hours (first stoke). After the first stoke, the samples were cooled in a desiccator. The samples were then added 1 mL of HNO_3 (2:1) and 0.5 mL of H_2O_2 (30%, Merck, Germany), and the hot plate- stoke procedure was repeated (second stoke). After the second stoke, the ash samples were cooled in a desiccator, dissolved in 5 mL of 10% HCl (36%, Centrom, Serbia), filtered through a quantitative cellulose filter paper (MACHEREY-NAGEL, Germany) in normal vessels of 25 mL and replenished with deionized water.

The leaching (migration) test was performed in order to investigate the possibility of the metals removal from the printed graphic product wastes (DN, P, T, C1, C2, CA, and C) in simulated environmental mediums. Printed graphic product waste samples (Kim et al., 2008) for test migrations were fragmented in pieces of the same size of 7.0 cm x 8.0 cm (total area: 56.0 cm²). All samples were immersed into 50 mL of the following leaching solutions: acidic (pH 3.25) and neutral (pH 7.0). This is a 120-day test and the analyses were carried out on days 1, 2, 3, 4, 5, 7, 10, 15, 20, 25, 30, 45, 60, 90 and 120 of the experiment at room temperature. For each measurement, 50 mL of aliquot was acidified with HNO_3 (65%, p.a., Merck, Germany).

The concentrations of metals in the solutions of printed graphic product wastes and the solutions of the leaching tests were determined by using an atomic absorption spectrophotometer (Thermo Scientific- Solaar S Series AA spectrometer), and the flame technique in accordance with 7000B EPA method (Environmental Protection Agency, 2007). The limits of detection for metals were: zinc 0.005 mg L⁻¹ (EPA 218.3), copper 0.040 mg L⁻¹ (220.1), chromium 0.025 mg L⁻¹ (EPA 218.3), cadmium 0.001 mg L⁻¹ (EPA 218.3), lead 1 mg L⁻¹ (EPA 239.1), and nickel 0.025 mg L⁻¹ (EPA 218.3). For the calibration curves, stock solutions containing 1000 mg of appropriate metal/mL (AccuStandard, Inc. USA) were used.

A sample of a municipal landfill was taken from the city landfill in Novi Sad. A sample of a illegal landfill was taken from the suburb of Kać (10 km away from Novi Sad, coordinates 45°18'09"N/19°56'19"E). The samples of the illegal and municipal landfills were dried at 105°C to constant weight. The concentrations of metals in samples

of the illegal and municipal landfills were determined after the acid digestion with HNO₃ (65%, p.a., Merck, Germany) in accordance with the 7000B EPA Method.

To compare the experimental data and the theoretical predictions of the impact of metals from the printed graphic product wastes on soil we also used the distribution coefficient, K_{sw} from literature data (Đukić, 2003), by using the concentration of metals leached in the neutral solution.

To evaluate the impact of the metal concentrations that have migrated from the printed graphic product wastes on land and water solutions, we used legislations:

1. The regulation on the emission limit values of pollutants in water and deadlines for achieving them, the emission limit values of wastewater disposal on the surface (Official Gazette RS 48/2012)
2. The regulation on the application of systematic monitoring of soil quality, indicators for assessing the risk of soil degradation and methodology for the development of remediation programs (Official Gazette RS 88/2010).

Results and discussion

The metal concentration levels in the printed graphic product wastes

The metal concentrations in the initial printed graphic product wastes are shown in Table 1. From the Table 1 it can be seen that chromium has not been detected in any of the printed graphic product wastes. The concentration of zinc ranged from 1.98 mg kg⁻¹ in the poster to 44.50 mg kg⁻¹ in the coffee package which was printed on the metalized polypropylene foil. The metal particles serve to create the silver glow or the gold-bronze effect. In order to achieve the metal effect, aluminum, copper and the alloys of copper and zinc (bronze) are usually used (Mumby, 2012; Prica et al., 2013).

The concentration of copper ranged from 4.35 to 44.70 mg kg⁻¹ and it cannot be said that there is a direct link between the increased copper concentration and the type of print used for the printing of the material in question. The elevated copper concentrations were found in toilet paper and cookie package 2 that were both printed by the flexo printing technique, but the same does not apply for the rest of the materials printed by the same technique (candy package, cookie package 1, coffee package). In the offset technique printed samples (daily newspaper and poster), copper concentrations did not vary in wide range (from 10.0 to 15.9 mg kg⁻¹). Cadmium concentrations varied in range from 0.26 mg kg⁻¹ in the toilet paper package to 0.65 mg kg⁻¹

in the candy package. Both of these two packaging materials have been printed by the flexo technique.

Table 1

The metal concentrations (mg kg⁻¹) in the printed graphic product wastes (Prica et al., 2014)

Printed graphic product waste	Concentration (mg kg ⁻¹)					
	Zn	Cu	Cr	Cd	Pb	Ni
Daily newspaper	10.60	10.00	<MDL	0.43	6.18	<MDL
Poster	1.98	15.90	<MDL	0.55	1.71	0.34
Toilet paper package	6.60	44.70	<MDL	0.26	<MDL	0.49
Cookie package 1	12.90	4.35	<MDL	0.43	1.18	<MDL
Cookie package 2	3.38	37.10	<MDL	0.53	<MDL	3.08
Candy package	6.20	11.30	<MDL	0.65	<MDL	1.03
Coffee package	44.50	6.85	<MDL	0.63	<MDL	3.28

The concentration of lead was the highest in the daily newspaper which was printed by the offset printing technique (6.18 mg kg⁻¹) and the lowest in the cookie package 1 (1.18 mg kg⁻¹) which was printed by the flexo printing technique. In the candy, toilet paper, cookie 2, and coffee packages, the lead concentrations have been below MDL.

The concentration of nickel varied in range from 0.34 to 3.28 mg kg⁻¹ and it can be concluded that in the materials that were printed by the offset printing technique a lower concentration of copper has been detected when compared to the materials that were printed by the flexo technique.

Table 2

Levels of metals in candy packages (Kim et al., 2008)

Manufacturing country (Sample no.)	Concentration (mg kg ⁻¹)				
	Cu	Cd	Pb	Cr	Zn
South Korea (31)	ND - 115.3	ND	ND - 9.9	ND - 44.2	ND - 114.8
China (39)	ND - 354.6	ND - 7.7	ND - 6394.1	ND - 1429.3	ND - 2579.5
Mexico (3)	10.9 - 66.1	ND	2.0 - 45.0	ND	2.0 - 9.4
Malaysia (3)	0.8 - 8.5	ND - 0.2	0.5 - 158.4	0.3 - 29.3	ND - 230.0
Indonesia (3)	3.7 - 529.8	ND	0.3 - 6.5	0.2 - 1.5	0.7 - 81.6
USA (2)	16.4 - 87.2	ND	ND	ND	3.2 - 74.3
Colombia (2)	3.3 - 33.1	ND	ND	ND	ND - 1.0
Others (9)	0.9 - 46.3	ND	ND - 13.7	ND - 82.6	0.2 - 102.6

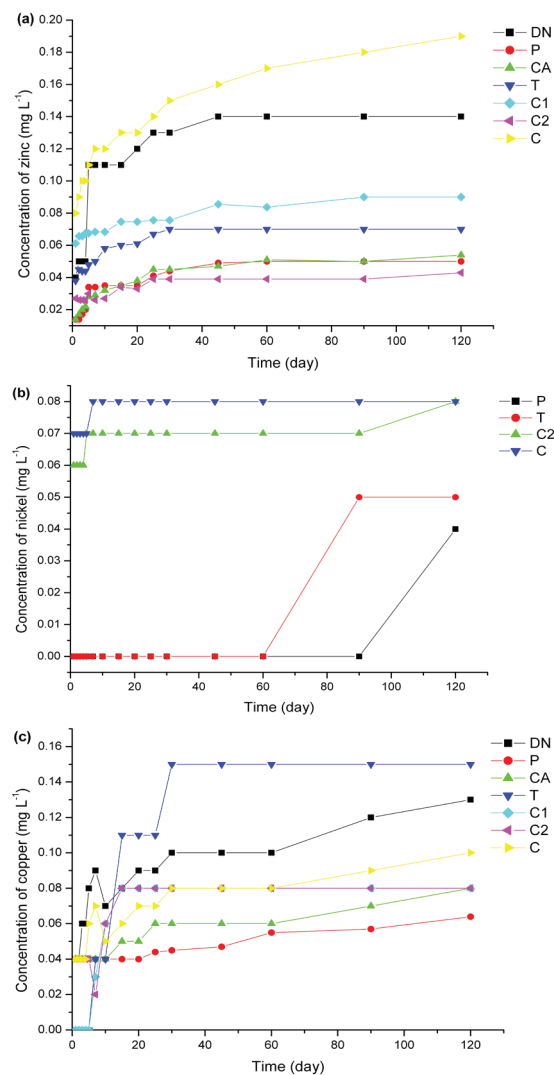
Note: ND - Not Detected

Table 2 shows the concentrations of metals detected in the candy packages made by various producers (Kim et al., 2008). By comparing the metal concentrations, we can conclude that they vary in extreme range and that in some of the packages the concentrations of metal are significantly higher than in the ones used in this study. It is obvious that the printing

techniques as well as the material used for the making of the final product influence the wide range of metal concentrations. The concentrations of copper in some of the graphic polymer materials even went as high as 530 mg kg⁻¹ (Kim et al., 2008), while the concentrations of zinc and copper in paper as well as in plastic graphic materials went up to 200 mg kg⁻¹ and 380 mg kg⁻¹, respectively (Long et al., 2011).

The leaching of metals from the printed graphic product waste to different pH value simulated solutions

Figure 1 shows the concentrations of the detected metals (zinc, nickel and copper) which migrated from various printed graphic product wastes (DN, P, T, C1, C2, CA, and C) in the neutral solution.



» **Figure 3:** The concentrations of (a) zinc, (b) nickel and (c) copper leaching from printed graphic product waste to a neutral solution

Cadmium, chromium and lead have not migrated from any of the investigated printed graphic product wastes.

The concentrations of zinc in all solutions were increased by increasing the time of contact between the printed graphic product wastes and the solution. In the acidic solution, the highest concentration of zinc has been detected, which is in accordance with literature data (Long et al., 2011). The leaching of zinc in the offset technique printed materials ranged from 0.05 to 0.16 mg L⁻¹, while it ranged from 0.01 to 0.23 mg L⁻¹ in the flexo technique printed materials, the candy package being the exception. After 90 days, the concentration of zinc varied in a very low percentage, except in the case of the candy package where a sudden increase has been detected (0.28 mg L⁻¹ after 90 days and 0.59 mg L⁻¹ after 120 days) when the neutral solution was used.

Nickel has not leached from the following printed graphic product wastes: daily newspaper, candy and cookie 1 packages. From the toilet paper package nickel has migrated to pH 3.25 and 7 solutions only after 90 days and even then in very low concentrations, from 0.04 to 0.05 mg L⁻¹, so that it can be concluded that the pH of the solutions did not have too much influence on the migration of nickel from the toilet paper. From the poster, the nickel migration has been detected after 20 days of the experiment (pH 3.25) as well as after 90 days (pH 7), but it had not changed significantly until the end of the experiment (from 0.04 to 0.09 mg L⁻¹). In the rest of the printed graphic product wastes, the concentration of nickel has also varied in a relatively narrow range from 0.06 to 0.09 mg L⁻¹, where the pH value of the solution did not have a significant influence.

The leaching of copper from some of the printed graphic product wastes in acidic solutions has been detected on the seventh day of the experiment (poster, toilet paper and cookie 1 packages). From all of the tested materials, the migration of copper ranged from 0.04 to 0.38 mg L⁻¹, where the pH value of the solution did not have a significant influence on the concentration level of copper in the leaching solution.

It cannot be clearly defined which printing technique or which printed graphic product waste contributes more to the leaching of metals. When compared to the emission limit values for the investigated metals regulated by the current Regulation (Official Gazette RS 48/2012), the concentrations of the said metals are below the limit values so that their disposal to surfaces, in this case soil, is allowed. The concentration of metals that are present in the tested printed graphic product wastes does contribute to a certain extent to the overall concentration of metals during disposal, but not in the concentrations that would be considered dangerous.

The assessment of metal (zinc, nickel and copper) concentrations in the soil of illegal and municipal landfills based on K_{SW} and metal concentrations in the leaching solution, which comes from the concentration of metals that have migrated from the printed graphic product waste, were calculated by the following equation (Đukić, 2003):

$$K_{SW} = \frac{c_s}{c_w} \quad (1)$$

where: K_{SW} - distribution coefficient ($L \text{ kg}^{-1}$), c_s - metal concentration in the soil (mg kg^{-1}) and c_w - metal concentration in the solution (mg L^{-1}).

The contribution of individual metals to the overall concentration in the soil after 120 days varied in the following range: Zn ($0.38 - 2.02 \text{ mg kg}^{-1}$), Ni ($0.14 - 0.31 \text{ mg kg}^{-1}$) and Cu ($0.18 - 0.51 \text{ mg kg}^{-1}$), which indicates that this concentration does not significantly influence the quality of soil. In Table 3, the concentration of zinc in the soil after the leaching from the deposited printed graphic product wastes to the soil of the illegal and municipal landfills has been calculated based on the concentration of the same metal that migrated from the suitable material to the neutral solution and distribution coefficients, K_{SW} taken from literature data (Đukić, 2003). After the calculated contribution of the zinc, nickel and copper concentrations leaching from the printed graphic product wastes to the soil and based on the Regulation on the application of systematic monitoring of soil quality, indicators for assessing the risk of soil degradation and methodology for development of remediation programs (Official Gazette RS 88/2010), it can be concluded that the concentrations are lower than those that would point to a significant contamination. In the initial samples of the soil of the illegal and municipal landfills there was a more significant difference between the concentrations of zinc (230.5 mg kg^{-1} for the illegal and 62.1 mg kg^{-1} for the municipal landfill) and the concentration of nickel (62.5 mg kg^{-1} for the illegal and 20.0 mg kg^{-1} for the municipal landfill), while the copper concentrations were approximately the same for both illegal and the municipal landfill (37.5 and 45.7 mg kg^{-1} , respectively). These initial metal concentrations have been below MDL which indicates that it is necessary to enforce remediation. After the theoretical calculation of the contribution to the concentration of metals in the soil of illegal and municipal landfills due to the leaching from the printed graphic product wastes, the concentrations also stay below the aforementioned MDL value.

During this calculation of the contribution to the overall concentration of metals due to the leaching from the printed graphic product wastes, many factors that are present in real conditions have not been included, and because of that disagreements with the experimentally

obtained results are possible. The concentration of metals certainly does depend on the physical and chemical nature of the soil because it determines the force of binding and forms of migration. The soils differ in contents and the capacity of metal binding, and the factors that influence the capacity of binding are numerous, and some of them should be singled out, such as: pH value, the overall cation adsorption capacity, the quantity and the type of clay, organic matter, iron, aluminum and manganese oxides, and redox potential (Vega et al., 2008).

Table 3

The assessment of zinc concentration (mg kg^{-1}) in the soil of illegal and municipal landfills based on K_{SW} and the zinc concentrations leaching from the printed graphic product wastes to the neutral solution

Landfills	Leaching time	Concentration (mg kg^{-1})						
		DN	P	T	C1	C2	CA	C
Illegal	1	230.9	230.6	230.8	231.0	230.7	230.6	231.2
	2	231.0	230.6	230.9	231.1	230.7	230.6	231.3
	3	231.0	230.6	230.9	231.1	230.7	230.7	231.4
	4	231.0	230.7	230.9	231.1	230.7	230.7	231.4
	5	231.5	230.8	230.9	231.1	230.8	230.7	231.5
	7	231.5	230.8	230.9	231.1	230.7	230.8	231.5
	10	231.5	230.8	231.0	231.1	230.7	230.8	231.6
	15	231.5	230.8	231.0	231.2	230.8	230.8	231.6
	20	231.6	230.8	231.0	231.2	230.8	230.8	231.7
	25	231.7	230.9	231.1	231.2	230.8	230.9	231.7
	30	231.7	230.9	231.1	231.2	230.8	230.9	231.9
	45	231.7	230.9	231.1	231.3	230.8	230.9	231.9
	60	231.7	230.9	231.1	231.2	230.8	230.9	232.0
	90	231.7	230.9	231.1	231.3	230.8	230.9	232.1
120	231.7	230.9	231.1	231.3	230.9	231.0	232.5	
Municipal	1	62.5	62.2	62.4	62.6	62.3	62.2	62.8
	2	62.6	62.2	62.5	62.7	62.3	62.2	62.9
	3	62.6	62.2	62.5	62.7	62.3	62.3	63.0
	4	62.6	62.3	62.5	62.7	62.3	62.3	63.0
	5	63.1	62.4	62.5	62.7	62.4	62.3	63.1
	7	63.1	62.4	62.5	62.7	62.3	62.4	63.1
	10	63.1	62.4	62.6	62.7	62.3	62.4	63.2
	15	63.1	62.4	62.6	62.8	62.4	62.4	63.2
	20	63.2	62.4	62.6	62.8	62.4	62.4	63.3
	25	63.3	62.5	62.7	62.8	62.4	62.5	63.3
	30	63.3	62.5	62.7	62.8	62.4	62.5	63.5
	45	63.3	62.5	62.7	62.9	62.4	62.5	63.5
	60	63.3	62.5	62.7	62.8	62.4	62.5	63.6
	90	63.3	62.5	62.7	62.9	62.4	62.5	63.7
120	63.3	62.5	62.7	62.9	62.5	62.6	64.1	

Conclusion

In the printed graphic product wastes, the presence of zinc, copper, cadmium, lead, and nickel has been detected and quantified, in a more or less wide range of concentrations depending on the printing techniques and the materials used. Based on the leaching test of metals from the tested printed graphic product wastes to solutions of different

pH value it can be concluded that the migration does occur, but the concentrations of metals that do migrate are not considered as dangerous when being disposed of according to the current Regulation (Official Gazette RS 48/2012). After the theoretical calculation of the contribution to the concentration of metals in the soil due to the leaching from the printed graphic product wastes, the concentrations remain below the concentration that would point to significant pollution (Official Gazette RS 88/2010). The results indicate that there has come to the increase in zinc and copper concentrations in the soil of the illegal and municipal landfills without the more significant difference in accumulation in regard to the soil of illegal and municipal landfills (to 5%). With all of the materials, there was an increase in the concentration of metals in the soil together with the increase of contact time between the printed graphic product wastes and soil. The contribution of zinc to the overall concentration of metals in the soil due to the leaching from the printed graphic product wastes is as follows: 1.70 mg kg⁻¹ (toilet paper package), 1.90 mg kg⁻¹ (cookie package), 2.10 mg kg⁻¹ (daily newspaper) and 2.40 mg kg⁻¹ (coffee package). The concentration of copper that could be considered as accumulation in the soil due to the migration from the deposited waste materials differs from the concentrations that were theoretically predicted: 1.40 mg kg⁻¹ (toilet paper package) and 1.90 mg kg⁻¹ (daily newspaper). The accumulation occurred after 10 days when the daily newspaper was used in the experiment, and in the experiment with the toilet paper package the accumulation of copper (as well as zinc) occurred only after 20 days of the experiment. Even with the determined contribution to the overall concentration of zinc and copper in the soil of illegal and municipal landfills, the printed graphic product wastes did not lead to a more significant soil contamination (Official Gazette RS 88/2010) at the applied experiment design. This experimental research is preliminary and in order to gain a more detailed insight into the assessment of the influence of these materials, further research is necessary, the one that would enlarge the number of parameters that would more realistically describe the state of things in landfills during a longer period of time, the one that would include a higher number of printed graphic product wastes, but also the one that would apply the obtained results to the realistic quantity of generated printed graphic product wastes.

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