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The Use of Municipal Solid Waste Compost in Contaminated Soil to Reduce the Availability of Ni and Cd: A Study from Istanbul

Emine Elmaslar Ozbaş

Department of Environmental Engineering, Faculty of Engineering, Istanbul University, Avcilar, 34320, Istanbul, Turkey; elmaslar@istanbul.edu.tr (for correspondence)

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This study investigated the short-term effect of adding Municipal Solid Waste (MSW) compost to artificially Ni and Cd polluted soil for a 3-month period. Before this study, the adsorption capacity of MSW compost to adsorb Ni and Cd was also investigated. At the end of the adsorption studies, it was found that Ni adsorption on MSW compost can be explained with the Freundlich model, and Cd adsorption with the Langmuir model. It was noted that in the short-term effect studies, the addition of compost to soil decreased the concentrations of the immediately available forms of Ni and *Cd.* The addition of compost to soil increased the potentially available form of Ni and Cd. The leachability of Ni assessed by the toxicity characteristic leaching procedure test increased with the compost amounts. However, the addition of compost did not affect the leachability of Cd. At the end of the sequential extraction procedure, it could be said that a 1% addition of compost increased the concentrations of Ni and Cd in organic matter bounded and residual fractions. The addition of more (>1%) compost, decreased the concentrations of Ni in these fractions. © 2015 American Institute of Chemical Engineers Environ Prog, 00: 000-000, 2015

Keywords: cadmium, compost, heavy metals, soil pollution, nickel

INTRODUCTION

The major risk of soil contamination with metals is their potential to leach into groundwater and threaten all forms of life. Metals persist in soils for a long time after their introduction into the environment, and most metals stand in soil without microbial or chemical degradation [1,2].

Municipal solid waste (MSW) generated in cities and urban areas is increasingly being recycled into agriculture throughout the world [3,4]. In Istanbul, about 14.000 tons of solid waste is generated daily [5]. Composting is a good alternative to landfilling and incineration. This is because composting reduces both CO_2 and atmospheric pollutant emissions [6].

The use of MSW compost improves the physical, chemical, and biological properties of soils by supplying organic matter (OM) [7]. Composts which contain a high proportion of humified OM, can decrease the bioavailability of heavy metals in soil by adsorption and by forming stable complexes with humic substances [8]. Both organic and inorganic contaminants have been successfully treated using compost, including pesticides, colorants, arsenic, and heavy metals such as lead, zinc, copper, and cadmium; even if high concentrations of these elements already existed in the compost [9]. The knowledge of metal speciation in compost-treated soils may be useful to quantify potential environmental effects. There is a common conception nowadays that the total concentrations of metals in soil are not good indicators of bioavailability, or good tools for potential risk assessment either [10,11]. For this reason, sequential extraction procedures were developed in order to predict retention, mobility, and bioavailability of trace metals in soils [12].

There is a large body of literature on the transfer of heavy metals in the composition of compost that is obtained from domestic solid wastes to plants [13–16]. Wu *et al.* [17] applied compost to rice plant in greenhouse environment and analyzed the uptake of Cd and Pb by the plants. The literature also contains studies involving EDTA assisted phytoextraction of heavy metals from MSW compost [18] and heavy metal removal from compost using zeolite [19]. Carbonell *et al.* [7] analyzed the transfer of heavy metals in compost by maize (*Zea mays* L.) plant and reported that Cu, Pb, and Zn levels increased after adding compost.

This study investigated the capacity of MSW compost to adsorb two toxic metals: Ni and Cd. MSW compost was artificially added to Ni and Cd contaminated soil in differing amounts, and the change of Ni and Cd concentrations in the soil (immediately available, potentially available) for a 3month period were examined. At the end of three months, a toxicity characteristic leaching procedure (TCLP) leaching test and sequential extraction procedure were performed on the soil samples (compost - added and not compost-added). In this way, the short-term effect of MSW compost on Ni and Cd fractionation was investigated.

MATERIALS AND METHODS

The MSW Compost samples used in this study were obtained from the Istanbul Solid Waste Recycling and Composting Facility, Turkey. The Commercial soil (brand of the soil; TROPIKAL Süs Bitkileri) was used as the soil sample. Acccording to the producer report, the soil structure is free from sand and other contaminations such as animal and plant residue.it has high lignin and cellulose structure. Nickel (II) nitrate-hexahydrate salt (99+% purity, ACROS Organics

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Table 1. Abbreviations of soil and soil-compost mixtures used in incubation experiments.

Abbreviation	Material			
S	Soil			
PS	Contaminated soil			
PS1	Contaminated soil +1% compost			
PS3	Contaminated soil + 3% compost			
PS6	Contaminated soil + 6% compost			

brand) and Cd nitrate $4H_2O$ (MERCK brand, Guaranteed Reagent suitable for use in analytical chemistry) were used in the experiments.

Characterization of Soil and Compost Samples

Distilled water was added to the compost samples at a 5:2 (v/w) ratio to determine the pH values of the samples. The pH values were measured using a pH meter (Jenway 3040 Ion Analyzer), after being mixed by a magnetic stirrer for 10 min [20].

The pH values of the soil samples were determined in a water suspension 1:2.5 sample: solution ratio and in KCl 0.1 N [21].

The TS 8340 [22] method was used to detect available P concentrations of the soil and compost samples; the TS 8341 [23] method was used for the detection of K concentrations, and the TS 8336 [24] method was used for the detection of organic substance contents (%) of the compost samples.

The percentages of C, H, N, and S in the compost and soil samples were determined in the Advanced Analysis Laboratory (IAL) at Istanbul University. Before the elemental analysis, the samples were dried at 50 °C for 24 h and ground to a fine powder in a quartz mortar. The equipment specifications were as follows: The Thermo Finnigan Flash EA 1112 Series Elemental Analysis device, temperature: 900 °C; mobile gas He; burning gas O_2 .

The soil and compost samples were air dried, sieved through a 2 mm mesh, and stored in polypropylene bottles for the determination of the total Cd and Ni concentrations. The "Microwave Solubilization Method" was used to determine the total metal concentrations of the soil and compost samples (EPA Method 3051A) [25]. According to this method, a 0.5 g homogenous dry sample was put in Teflon containers. Then, ultra-pure 9 mL HNO3, 3 mL HCl, and 2 mL HF were added, and the Teflon containers were placed in the microwave (Berghof MWS-2 microwave-system). They were gradually heated up to 185 °C in the microwave and kept at this temperature for 10 min. The samples in the Teflon containers were filtered (MN 640 de, 125 mm Macherey-Nagel filter paper), put into HDPE volumetric flasks, and then ultra-pure water was added until it reached 50 mL. Cd and Ni concentrations were measured with a Hach-Lange DR 5000 photometer (LCK 308 kit with a 0.02-0.3 mg/L measuring range for Cd and a LCK 337 kit with 0.1-6 mg/L measuring range for Ni). Replicated measures of a reference material (NCS Certified Reference Material, NCS ZC73002 Soil) were used to assess contamination and precision. The analytical precision that measured as relative standard deviation was routinely between 5 and 6%.

Adsorption Studies

Batch experiments were performed to determine the adsorption isotherms of Cd and Ni within the compost. In experiments determining the effect of time on adsorption, 80 mL was taken from each of 75 mg/L Ni solution (prepared from Nickel (II) nitrate-hexahydrate salt) and 3 mg/L Cd solution (prepared from Cd nitrate $4H_2O$) and added to 3 g

of composts. The samples were shaken with horizontal orbit shakers at 25° for 5 min 24 h at 120 rpm. In studies conducted to determine the effect of concentration on adsorption, 80 mL of Ni solutions prepared with different concentrations was added to 3 g of compost samples and shaken at 25° for 2 h in 120 rpm. The initial pH of the suspensions was not modified during the experiments to simulate real environmental conditions of compost application. The final pH in the extracts was recorded. After the adsorption, samples filtered from 0.45 μ m membrane filter were diluted. Ni and Cd concentrations in filtrates were measured with a Hach-Lange DR 5000 photometer.

The two most common models (Freundlich and Langmuir models) to describe adsorption data have been appropriately applied to the experimental data. The Langmuir theory can be represented by the following expression (Eq. 1) [26]:

$$(C_{\rm e}/q_{\rm e}) = (1/q_{\rm m}K) + (1/q_{\rm m})C_{\rm e}$$
⁽¹⁾

where $q_{\rm m}$ is the maximum amount of adsorption (mg/g), *K* is the affinity constant (L/g), and $C_{\rm e}$ is the solution concentration at equilibrium (mg/L). The well-known logarithmic form of the Freundlich model is given by the following equation (Eq. 2) [26]:

$$\ln q_{\rm e} = \ln K_{\rm F} + (1/n) \ln C_{\rm e} \tag{2}$$

where $K_{\rm F}$ and *n* are the Freundlich constants related to the adsorption capacity and adsorption intensity, respectively. Thus, the plot of $\ln q_{\rm e}$ against $\ln C_{\rm e}$ of the Eq. 2 should provide a linear relationship whereby 1/n and $K_{\rm F}$ can be determined from the slope and the intercept, respectively.

Incubation Studies

Compost and soil samples were mixed manually. These samples were put in 500 mL plastic pots; 200 mL of solution comprising 75 mg/L Ni and 3 mg/L Cd was added to these pots. Samples within the pots were mixed well. These pots were kept at 25 °C under laboratory conditions for 90 days. Their moisture was maintained throughout the experiment around 76% of their field capacity. Two pots were used for each compost-soil mixture. Abbreviations used for soil and soil-compost mixtures are presented in Table 1. At the end of every 30 days, composite samples were taken from each pot and the pH was determined for air-dried samples. pH determination was conducted with both methods mentioned above. Concentrations of immediately available elements and potentially available elements were determined for Ni and Cd. At the end of 90 days, a TCLP leaching test was performed on composite samples taken from pots following the air-dried. In addition, sequential extraction was performed on these samples and metal types (for Ni and Cd) were determined.

Immediately Available Elements Determination Method

To determine the immediately available elements, the concentrations for Ni and Cd, 2 g were taken from each of the air-dried samples. For these samples, 20 mL was added from 0.01 N CaCl₂ solution, and then shaken for 3 h. Extracts filtered through a 0.45 μ m membrane. Ni and Cd concentrations in filtrates were determined [9].

Potentially Available Elements

In the determination of potentially available elements, 50 mL of the solution with 4.95 pH and comprising 0.5 M ammonium acetate and 0.02 M EDTA was added to 5-g airdried samples. After shaking for 1 h, they were centrifuged at 700 rpm for 15 min. Extracts were filtred through a 0.45 Table 2. Soil and compost main properties.

Parameters	Soil	Compost		
pН	5.5–6	7.9		
P (mg/kg)	15	3.59		
K (mg/kg)	70	4.87		
C%	10.08	9		
N%	0.26	1		
C/N	38.77	9		
H%	9.56	1.22		
S%	_	_		
OM (%)	1.83	1.36		
Cd (mg/kg)	ND	1		
Ni (mg/kg)	ND	28		



Figure 1. Time dependent change of removal efficiencies in adsorption experiments and change of pH values following the adsorption.

 μ m membrane filter. Then, Ni and Cd concentrations in filtrates were determined [9].

TCLP Leaching Test

The EPA Method 1311 was used for the TCLP leaching test. Soil samples and distilled water (the pH value adjusted to 4.93 with acetic acid) were mixed at a ratio of 1:10 and shaken at 25 °C for 18 h at 30 rpm. After the extraction, the mixture was centrifuged at 2000 rpm for 15 min. Then, the extracts were filtered through a 0.45 μ m membrane filter. Determination of Ni and Cd was made on these filtrates [9].

Sequential Extraction Method

The heavy metal sequential extraction, which has been used successfully in soils amended with compost [27,28], included the following steps:

- 1. 0.1 M CaCl₂ (1:10, w/v) for 16 h: metals in soil solution and in exchangeable forms.
- 2. 0.5 M NaOH (1:10, w/v) for 16 h followed by aqua regia digestion: metals associated with OM.
- 3. 0.05 M Na₂H₂EDTA (1:10, w/v) for 1 h: metals which can be extracted by a strong chelator.
- 4. Digestion with aqua regia: residual metals.

Ni and Cd concentrations of extracts obtained at each stage were measured with a Hach-Lange DR 5000 photometer.

All physico-chemical analyses were duplicated.

RESULTS AND DISCUSSION

Characterization of Soil and Compost Samples

The characterization of the soil and compost samples is presented in Table 2. It was shown that the pH of the soil sample was close to a neutral pH with slightly acidic charac-



Figure 2. Changes of removal efficiencies depending on the initial metal concentrations in the adsorption experiments.

teristics. Ni and Cd were not detected in the original soil samples. According to the measurements, the Ni amounted to 28 mg/kg while the Cd amounted to 1 mg/kg in the compost.

Adsorption Studies

According to the results of adsorption experiments conducted with time, it was found that MSW compost adsorbed Ni and Cd within aqueous solutions well. An 80% removal efficiency of Ni and a 100% removal efficiency of Cd were obtained at the end of 120 and 130 minutes, respectively (Figure 1). It was recorded that in adsorption experiments, as the Ni concentration of the solution increased, the removal efficiency increased. In experiments on Cd, it was observed that as the concentration increased, the removal efficiency decreased. However, around 84% removal efficiency was obtained even in a 10 mg/L Cd concentration. At the beginning of the adsorption periods, pH values of the Ni and Cd solutions were 4.5 and 5, respectively. These values reached 6.5 following the adsorption at the end of 1440 min (24 h) (Figure 1). In the adsorption experiments, it was determined that as metal concentrations within aqueous solution increased, the pH values of the solution following the adsorption did not change. After the adsorption, the pH values measured 6 for Ni solutions and 5.5 for Cd. Similarly, Cay et al. [29] reported that pH value of Cd adsorption was 5.5, which is close to the pH of the original solution. As reported in the study of Malkoc and Nuhoglu [30], an increase in pH may result from the interactions between Ni(II) and Cd(II) ions and functional groups of MSW compost. As reported in the study of Paradelo and Barral [9], the change in solution concentrations affects adsorption of various materials differently.

It was found that as initial metals concentrations within aqueous solution increased, removal efficiencies for Ni increased, but removal efficiencies for Cd decreased (Figure 2).

At the end of the isotherm studies, it was found that Ni adsorption on MSW compost can be explained with the Freundlich model (R^2 = 0.981) and Cd adsorption can be explained with the Langmuir model (R^2 = 0.9932; Table 3). In the Freundlich model, the n constant refers to adsorption intensity. As the *n* value increases, affinity will be higher. In addition if n = 1, adsorption is linear. If n < 1, it can be observed that the adsorption process is a chemical process and if n > 1 it can be taken that the adsorption process is realized via the physical process. According to the results of this study (Table 3), we can say that chemical processes are effective on Ni adsorption. Srivastava *et al.* [31] reported that similar adsorption mechanisms are effective for Ni. In this study, it was found that maximum adsorption capacity of MSW compost was 7.24 mg/g for Cd (Table 3). In studies on

Ni and Cd adsorption in the literature, it was found that the maximum sorption capacity for Ni onto activated sludge was 17.17 mg/g [32] and the adsorption capacity of clinoptilolite for Ni was 0.5–0.9 mg/g [33]. In studies conducted with bag-gase fly ash, it was found that adsorption capacity was 0.95–5.78 mg/g for Ni and 0.89–5.18 mg/g for Cd [31].

Table 3. The Langmuir and Freundlich constants and correlation coefficients of isothermmodels for the adsorption of Ni and Cd from aqueous solution onto the composts.

Freundlich	Ni	Cd	
$\overline{K_{\rm f}({\rm mg}^{1-1/n}{\rm L}^{1/n}{\rm g}^{-1})}$	0.1997	0.0217	
N	0.666	2.602	
R^2	0.981	0.1316	
Langmuir	Ni	Cd	
$K_{\rm L}$ (L/g)	28.547	0.124	
$q_{\rm m} ({\rm mg/g})$ R^2	-	7.24	
\hat{R}^2	0.3136	0.9932	

Incubation Experiments

During the incubation process, it was noted that the pH values of all the samples tended to decrease with time. In the pH measurements performed at the end of every month, it was seen that the decrease of the soil pH tamponed with the increase of added composts, and remained around the original 5.5 pH level (Table 4). Similar results were obtained in studies conducted by [21,28]. Today, in many countries' legislation, it is stated that the pH value of soil should be 6 or above [34]. In this study, employing used compost, it was found that the pH value of soil was around 6 at the end of the 3-month incubation period.

Availability Analysis

In experiments with immediately available elements, no Cd was detected in the soil samples (PS1, PS3, and PS6) at the end of the first month and following months. As for Ni amounts, it was determined that at the end of second month, immediately available Ni amounts of the soil samples (PS1, PS3, and PS6) fell below half the amount of the first month, and increased at the end of the third month (Table 5).

Table 4. The change of pH values measured with distilled water and KCl solution.

Treatment	At the end	of first month		nd of second nonth	At the end of third month		
	pH (water)	рН (0.1 N KCl)	pH (water)	рН (0.1 N KCl)	pH (water)	рН (0.1 N KCl)	
S	6	5.5	5.8	5.3	5.6	5.2	
PS	5.61	5.15	5	5.37	5	5.5	
PS1	6.3	6.1	5.7	5.17	5	5.5	
PS3	6.55	6.25	6.34	6	5.2	5.8	
PS6	6.49	6.45	6.6	6.87	5.5	6	

Table 5. The results of immediately available elements experiments.

Immediately available elements									
	At the end of	f first month	At the end of	second month	At the end of third month				
Treatment	Cd (mg/kg)	Ni (mg/kg)	Cd (mg/kg)	Ni (mg/kg)	Cd (mg/kg)	Ni (mg/kg)			
S	ND	ND	ND	ND	ND	ND			
PS	1.42	11.05	ND	3.04	ND	5.43			
PS1	ND	9.37	ND	5.39	ND	13.9			
PS3	ND	9.1	ND	2.8	ND	11.8			
PS6	ND	7.9	ND	4.9	ND	10.3			

ND: Not Detected.

Table 6. The results of potentially available elements experiments.

Potentially available elements									
	At the end o	f first month	At the end of	second month	At the end of third month				
Treatment	Cd (mg/kg)	Ni (mg/kg)	Cd (mg/kg)	Ni (mg/kg)	Cd (mg/kg)	Ni (mg/kg)			
S	ND	ND	ND	ND	ND	ND			
PS	0.47	2	1.516	0.19	1.386	0.392			
PS1	0.74	0.44	0.045	0.395	0.617	0.731			
PS3	1.46	0.953	1.47	0.793	0.69	1.07			
PS6	1.4	1.55	1.39	1.58	0.61	1.59			

ND: Not Detected.

Comparing the results of [21] and this study, the results of Cd in this study are similar to the tendency observed for immediately available concentrations of Cu; and the tendency observed for immediately available concentrations of Pb and Zn is similar to the results of the Ni in this study. In the soil samples that added 6% composts (PS6), it was seen that at the end of the third month, immediately available Ni concentrations were higher than the values of the first month. It is assumed that this resulted from the fact that the Ni within the compost started to decompose in the soil at the end of 3 months. It was found that the addition of compost generally decreased immediately available Ni and Cd amounts in the soil—this effect lasted for 2 months for the Ni.

Potentially available Ni and Cd concentrations in soil samples increased as the addition of compost amounts increased. It was detected that at the end of the second month, potentially available Cd concentration in PS1 decreased 93% and increased to the amount of the first month at the end of the third month. It was detected that in PS3 and PS6 samples, potentially available Cd concentrations did not change at the end of two months and decreased 57% at the end of the third month. It was detected that potentially available Cd concentrations in PS samples tripled at the end of the second month. It was detected that Ni concentrations in PS samples did not change for three months; potentially available Ni concentrations in PS1 and PS3 samples decreased at the end of the second month and increased again at the end of the third month. Potentially available Ni concentrations in PS6 samples did not change for three months, and were higher (1.55-1.59 mg/kg) compared to the other soil samples (Table 6).

The results of TCLP leaching revealed that as the amount of compost in the soil samples increased, leachable Ni amounts increased as well (from 12.3 to 27.3 mg/kg). Regarding Cd amounts, no leachable Cd concentration was

	At the end of third month					
Treatment	Cd (mg/kg)	Ni (mg/kg)				
S	ND	ND				
PS	ND	12.3				
PS1	ND	17.65				
PS3	ND	22.65				
PS6	ND	27.3				

Table 7. The results of TCLP leaching test.

*ND: Not Detected.

detected at the end of three months (Table 7). It was found that the compost added to soil decreased mobile form concentrations of Ni and Cd in soil and this effect increased by the increase in the compost amounts. Comparing the results of [21] and the present study, TLCP test results on Cu, Pb, and Zn are similar to the results of this study.

Experiments conducted with powerful complexing agents such as EDTA (e.g., potentially available experiments) are used to determine organically bound phases of elements in soil and compost. Experiments conducted with neutral salts such as $CaCl_2$ are used to determine weakly bound elements in soil [21]. This study reveals that Cd is organically bound and Ni is weakly bound (Tables 5 and 6).

Regarding the Ni and Cd fractions following the sequential extraction, it was detected that the concentrations of Ni and Cd in the OM bounded and residual fractions were higher compared to the concentrations in other fractions. Comparing soils both with and without compost, it was observed that as the compost amount increased, the concentrations of the Ni in CaCl2, and OM bounded, residual fraction decreased. It was detected that especially with the addition of 6% compost, there was a 50% decrease in CaCl₂ and OM bounded fractions of Ni (it was found that Ni concentration decreased to 10.97 from 24.04 mg/kg for CaCl₂ fraction, concentration in OM bounded fraction fell to 25.02 from 47.8 mg/kg). However, it was detected that the concentration of Ni fractions that can be extracted with strong chelator increased as compost amounts added to soil increased (from 4.55 to 44.68 mg/kg; Table 6). It was detected that Cd was found in OM bounded (3.3-3.48 mg/kg) and residual fractions (3.29-3.53 mg/kg) and it was not detected in other fractions. It was observed that Cd concentrations did not change with the increase in compost amounts added to the soil (Table 6). Contrary to the present study, [12] reported that the addition of MSW compost to soil affects concentrations of the Cd fractions as well and does not cause any change in concentrations of Ni fractions. It is assumed that the difference between these two studies results from the different characterization of soil samples (Table 8).

CONCLUSION

At the end of this study, it can be concluded that chemical processes are effective on the Ni adsorption and maximum adsorption capacity of MSW compost at 0.843 mg/g for Ni and 7.24 mg/g for Cd.

It was observed that the addition of 1, 3, and 6% MSW compost to soil tamponed the pH value of the soil, and thus the pH value did not fall below the original 5.5–6 pH value.

In this study, it was found that the addition of compost to soil decreased the concentrations of immediately available

Table 8. Metal concentrations (mg/kg dry soil) in the treatments determined by sequential extraction and total metal concentrations.

Treatment		aCl ₂ actable	Metals ass. with OM		Metals, which can be extracted by a strong chelator		Residual metals		Total metals	
	Cd	Ni	Cd	Ni	Cd	Ni	Cd	Ni	Cd	Ni
S	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
PS	ND	24.04	3.2	47.8	ND	4.55	3.42	9.08	6.68	85.47
PS1	ND	14.38	3.48	54.07	ND	7.34	3.29	9.24	6.78	85.03
PS3	ND	12.9	3.44	44.37	ND	18.84	3.43	9.19	6.89	85.67
PS6	ND	10.97	3.3	25.02	ND	44.68	3.53	7.53	6.85	88.2

ND: Not Detected.

forms of Ni and Cd. In potentially available elements experiments, it was observed that the addition of compost to soil increased this form of Ni. It was observed that the concentration of the potentially available form of Cd increased with the compost amounts; however, Cd concentrations in this form were 50% less than soil samples without compost at the end of 3 months. The leachability of Ni assessed by the TCLP test increased with the compost amounts. The addition of compost did not affect the leachability of Cd. In this study, the biggest advantage of the addition of compost was the fact that it decreased the metal concentration of the immediately available form, which is one of the most active forms of metals.

Results of the sequential extraction revealed that the addition of 1% compost increased the concentrations of Ni and Cd in OM bounded and residual fractions. The addition of more especially 6% compost decreased concentrations of Ni in these fractions and increased concentration in fraction that can be extracted with a strong chelator.

To conclude, the use of compost prevents easy decomposition of Cd and Ni in soil and thus protects the environment against such a danger. However, it is assumed that using the MSW compost at high rates such as 6%, may lead to negative results overshadowing the benefits mentioned above.

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