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Posted Date: 8 January 2025

doi: 10.20944/preprints202501.0575.v1

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## Article

# Retention of Copper and Zinc from Traffic Area Runoff by Topsoil of Vegetated Infiltration Swales Amended with Recycled Demolition Waste

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**Abstract:** Infiltration swales are a prospective key component of water-sensitive urban planning. The utilization of appropriate soil amendments is intended to facilitate their retention of pollutants from stormwater runoff of traffic areas. Little is known about the possibility of utilizing processed construction and demolition waste (CDW) as an amendment to improve pollutant retention. We conducted batch and field tests to investigate i) the leaching of metals from soil substrates containing CDW and ii) their retention potential for copper (Cu) and zinc (Zn) when charged with real traffic area runoff. To gain a comprehensive understanding of the chemical interactions, we iii) employed sequential extractions using an optimized protocol from treated and untreated soil substrates. In batch tests, the potential of vanadium leaching from technosols amended with brick-dominated CDW was apparent. When charged with traffic area runoff, the retentions of Cu and Zn in the technosols were comparable to those of the control soil without CDW. However, the simulation of high rainfall intensities reduced Cu and Zn retention in the technosols and the control. The results from the subsequent sequential extraction of Cu and Zn imply shifts in the chemical binding in the technosols compared to the control.

**Keywords:** technosols; stormwater management; green infrastructure; waste management; engineered soils; groundwater protection; pollutant retention

## 1. Introduction

Traffic area runoff (TAR) constitutes a substantial source of pollution, transporting a diverse array of metals to the environment [1,2]. The metals copper (Cu), and zinc (Zn) from brake dust and tire wear, respectively, are expected to have the highest concentration in TAR [3,4]. An effective solution to manage metal contamination is the implementation of roadside infiltration swales, which can be integrated into stormwater management strategies and is the state of the technology in Germany [5].

Recently, there has been growing interest in using construction and demolition waste (CDW), which consists mainly of concrete and bricks, as an innovative, cost-effective material for runoff treatment [6,7]. CDW is one of the most significant waste streams in Europe, and its utilization could reduce CO<sub>2</sub> emissions and save landfill space [8,9].

In this context, the concept of technosols, or human-made soils created from the recycling of materials such as CDW, has gained attention as a key approach to promoting sustainability within the framework of the circular economy [10]. In landscaping, the development of local material flows is key to a sustainable construction sector, and local processing and use of CDW can contribute to this [11]. Repurposing CDW as a topsoil amendment in infiltration swales offers benefits: reducing the environmental burden of waste disposal and providing an efficient means of mitigating road

runoff pollutants. However, the use of CDW is often hindered by issues related to acceptance and concerns about the release of potentially hazardous substances [12,13].

This three-year study investigates the potential of six CDW-derived technosols to remove Cu and Zn from genuine traffic runoff on a pilot scale for the first time compared to typical soil used in infiltration swales as a control. By examining the material composition-dependent leaching of substances based on batch tests and retention efficiency for Cu and Zn from real traffic area runoff under semi-real conditions, this research aims to evaluate their viability as an alternative to traditional filtration systems.

We hypothesize that topsoil from infiltration swales can be enriched with CDW without harming groundwater quality. In addition, we hypothesize that pollutant retention from traffic area runoff can be improved if the topsoil is enriched with CDW. We also hypothesize differences in the chemical binding of Cu and Zn in the technosols compared to typical soils used for infiltration swales.

To test the hypotheses, we investigated three-step phases:

Phase 1: Batch tests of different technosols (soil mixtures based on CDW) and the evaluation of the potential groundwater hazard

Phase 2: A field test of the technosols on the retention potential and efficiency of Cu and Zn when charged with real traffic area runoff

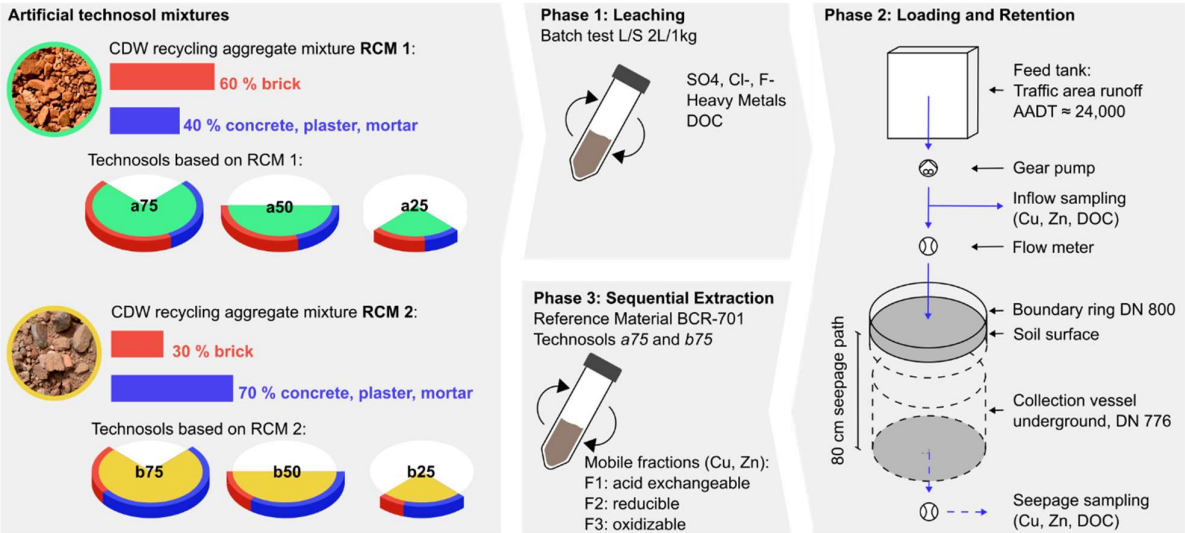
Phase 3: Sequential extraction using reference material BCR 701 (BCR-SEP) of Cu and Zn from the technosols with the highest CDW ratios after treatment with TAR to understand binding mechanisms.

The findings could contribute to developing sustainable solutions for urban pollution control while simultaneously addressing waste management challenges and advancing the circular economy.

2. Materials and Methods

2.1. Technosols from CDW

For the investigations, different technosols were produced based on two recycling mixtures (RCM; RCM1 and RCM2) that derive from building demolition and processing its CDW. The RCMs are crushed aggregates with grain sizes of 0 to 16 mm. RCM1 is enriched with brick material. It contains 60 % bricks and 40 % concrete, plaster and mortar. RCM2 is enriched with concrete, plaster, and mortar (70 %) and contains 30 % bricks (Figure 1). These RCMs have been blended with natural topsoil, subsoil, and compost to achieve comparable particle size distributions and organic matter contents (Table 1). The resulting six technosols ((a75, a50, a25, b75, b50, b25) containing different CDW ratios and compositions and one control soil (ctl) that did not contain CDW were produced.



**Figure 1.** Schematic representation of the study design.

**Table 1.** Technosol characteristics (as partly published in Knoll, et al. [14]; GWC = green waste compost; TS = topsoil; SS = subsoil,  $k_f$  = hydraulic conductivity; n.a. = not analyzed.

Sample	CDW	Material ratios [% v/v]				Grain size distribution [% w/w]				k <sub>f</sub> [m/s] ± SD	CaCO <sub>3</sub> [% w/w]	pH before treatment	pH after treatment
												with TAR	with TAR
		Bricks	Concrete + mortar + plaster	GWC	TS + SS	Clay	Silt	Sand	Gravel				
a75	RCM1: 75 %	41	27	10	22	9.6	13.8	40.7	35.9	1.8x10 <sup>-3</sup> ± 1.7x10 <sup>-4</sup>	45.3	7.5	8.0
a50	RCM1: 50 %	28	18	10	44	9.5	12.0	43.2	35.3	1.0x10 <sup>-3</sup> ± 7.6x10 <sup>-5</sup>	40.7	7.5	n.a.
a25	RCM1: 25 %	14	9	10	67	9.1	12.2	43.4	35.2	1.1x10 <sup>-3</sup> ± 8.1 x10 <sup>-5</sup>	29.3	7.5	n.a.
b75	RCM2: 75 %	20	48	10	22	8.9	12.4	42.1	36.6	2.2x10 <sup>-4</sup> ± 1.4x10 <sup>-5</sup>	53.7	7.8	8.2
b50	RCM2: 50 %	14	32	10	44	9.5	12.2	41.3	36.9	2.8x10 <sup>-4</sup> ± 1.6x10 <sup>-5</sup>	46.7	7.8	n.a.
b25	RCM2: 25%	7	16	10	67	10.5	11.3	42.9	35.3	3.1x10 <sup>-4</sup> ± 1.7x10 <sup>-5</sup>	34.6	7.7	n.a.
ctl	-	-	-	10	90	9.3	11.4	43.7	35.7	9.6x10 <sup>-4</sup> ± 5.6x10 <sup>-5</sup>	22.3	7.8	7.6

2.2. Study Design

For lysimeter tests, seven vessels (Polyethylene, DN 776 mm, height = 500 mm) were buried 80 cm below ground in 2021 and filled with the six different technosols and *ctl* (see **Error! Reference source not found.**). An extensive grass mix was then sown over the surface areas of all lysimeters to establish the vegetation. Boundary rings were placed over the surfaces of the vessels (High-Density Polyethylene, DN 800). A plastic hose connected the outlets (1/2”) at the bottom of the vessels to manholes where the seepage was quantified and collected using tipping counters (100 mL, polycarbonate, REED-Sensor, max. flow rate = 5 L/min).

The assessment considered two thresholds: German threshold values for seepage water (TV\_1) and for soil solution (TV\_2) according to the German Federal Soil Protection and Contaminated Sites Ordinance (Bundesbodenschutz- und Altlastenverordnung; BBodSchV) [15].

**Phase 1 – Leaching behaviour**

To assess the leaching of substances for the pathway soil-groundwater in advance, laboratory batch tests according to DIN 19529 with a liquid-to-solid ratio of 2 L/kg batch aqua test were conducted with RCM1, RCM2, the six technosols, and the *ctl* in triplicate [16]. Obtained eluates were analyzed for selected parameters according to the threshold values TV\_2 for soil solutions.

Besides total Cu and Zn concentrations in the eluate, the investigated parameter spectrum included the total concentrations of the metals Sb, As, Pb, B, Cd, Cr, Cr(VI), Co, Mo, Ni, Hg, Se, Tl, V, and Sn. Additionally, sulfate, chloride, fluoride, and dissolved organic carbon (DOC) were analyzed to evaluate potential risk for groundwater contamination (List of methods, limits of quantification (LOQ) and threshold values see Table A1).

**Phase 2 – Retention of Cu and Zn from traffic area runoff**

After backfilling the lysimeters, the six technosols and the *ctl* were exposed to natural weather conditions to ensure stabilization until the Cu and Zn discharges had normalized to avoid biasing

seepage concentrations when charging with TAR and measuring background concentrations. The quantities of seepage water up to the start of the TAR charging were recorded using tipping counters and reported as L/S in the results section. Mean background concentrations of Cu, Zn, V, and DOC are based on the values of 4 random samples per technosol between June 2023 and February 2024.

From April 2024 to August 2024, the lysimeter vessels were charged 12 times (6 per rainfall intensity) with TAR, collected at a highly trafficked road (approx. 24,000 vehicles/day; [17]). The TAR was transported to the study site, stored in containers, and pumped to the test plots using gear pumps. The flow rate was measured using flow meters. Two rainfall intensities were applied according to measured data from the German Weather Service (KOSTRA data): RI\_1: 121 L/(s·ha), volume per charging = 139 L, and RI\_2: 221 L/(s·ha), volume per charging = 127 L. Charging volume and flow rate of TAR were calculated according to the German guideline DWA-A 138-1, assuming a ratio of 15 m<sup>2</sup> of asphalt surface (mean runoff coefficient = 0.9) to 1 m<sup>2</sup> of active infiltration area in a swale. Influent and total seepage of the lysimeters were sampled and analyzed for Cu and Zn concentrations after aqua regia (AR) digestion and compared to the threshold values TV\_1 for seepage water

**Phase 3 – Sequential extraction of Cu and Zn from soil**

Soil samples from *a75*, *b75*, and *ctl* were taken in triplicate from the infiltration areas and, as a control, from the non-infiltration areas at depths of 0 - 25 mm. From these soil samples, Cu and Zn concentrations were further analyzed following the three-step optimized sequential extraction protocol (BCR-SEP) using BCR 701 (Community Bureau of Reference, European Commission) reference material according to Rauret, et al. [18]. All used utensils were made of borosilicate glass, polypropylene, or polytetrafluoroethylene (PTFE). The extractions were carried out in 80 mL centrifuge tubes (Herolab, Wiesloch, Germany), which were cleaned with 4 mol/L HNO<sub>3</sub> before use and then rinsed with ultrapure water [19]. From each sample, 1 ± 0.01 g was weighed into the centrifuge tubes three times. Additionally, as a reference BCR-701 sediment (European Commission, Joint Research Center, Belgium) was added to each batch for quality control. The individual steps of the procedure, including the different fractions and chemical solutions, are briefly described in Table 2. Calculated total Cu and Zn contents are the sum of fractions S1 to S4.

**Table 2.** Four steps of the optimized BCR-SEP with corresponding fractions and solutions [18].

Step	Fraction	Solution	Process description
S1	Acid extractable	40 mL acetic acid (0.11 mol/L)	Addition of the solution to the samples in centrifuge tubes;
			Overnight extraction (16 ± 2 h) in an end-over-end shaker at 30 ± 10 rpm and 21 ± 2°C;
			Centrifugation at 4000 g for 20 min to separate the extract from the technosol;
			Pipetting off the supernatant, filtering with 0.45 µm, and stabilizing with 50 µl of 65% HNO <sub>3</sub> ;
			Washing the residue with 20 mL of ultrapure water, shaking for 15 min, centrifuging for 20 min, pipetting off, and discarding the supernatant
S2	Reducible	40 mL hydroxylammonium chloride (0.5 mol/L)	Addition of the solution from a 1-L mixture (containing 25 ml of 2 mol/L HNO <sub>3</sub> ) to the residue;
			Continuation of the procedure as described in S1
S3	Oxidizable	2x 10 mL hydrogen peroxide (8.8 mol/L); 50 mL ammonium acetate (1 mol/L)	Addition of 10 ml H <sub>2</sub> O <sub>2</sub> to the residue, covering the tubes, and reaction
			at 21 ± 2°C for 1 h with occasional manual shaking;
			Placement of the tubes in a water bath (85 ± 5°C), reduction of the volume to <3 mL (occasional manual shaking);
			Addition of 10 mL H <sub>2</sub> O <sub>2</sub> (tubes in the water bath) and reaction until the volume is reduced to 1 mL (occasional manual shaking);
			Addition of 50 ml NH <sub>4</sub> OAc (pH 2) and continuation of the procedure as described in S1



S4      Residue      HNO<sub>3</sub>: HCl = 3:1      Digestion of the residue with AR

Due to difficulties with separation during centrifugation, the speed was increased from 3000 g to 4000 g. To compare the total concentrations from S1 to S4, an AR digestion was performed directly in each batch with 3 ± 0.1 g per technosol plus a BCR-701 reference. The Zn and Cu concentrations in the extracts were determined using inductively coupled plasma optical-emission spectrometry (ICP-OES).

In addition, all technosols’ dry weight and loss on ignition (LOI) were determined. For this purpose, 1 ± 0.01 g of each technosol in triplicate and one BCR-701 reference per batch was weighed and dried overnight at 105 ± 2 °C in an oven until a constant weight was achieved. These values were used as correction factors, and all analytical results in this work refer to the dry mass of the samples. To determine the LOI, the samples were heated in a muffle furnace at 550 °C for 2.5 h.

2.3. Data Processing

For comparisons of means, p-values were calculated using the Mann-Whitney U test with Python’s scipy package [20]. The data was processed with Python’s pandas [21], and the plots were created with Python’s matplotlib [22] and seaborn [23].

3. Results

3.1. Leaching Potential

The concentrations, pH values, and EC of the leached substance in the batch tests are summarized in Table 3.

**Table 3.** Substance concentrations in the eluate of the technosols *a75*, *a50*, *a25*, *b75*, *b50*, and *b25*, the control (*ctl*), and the two CDW mixtures RCM1 and RCM2 based on batch tests at L/S ratio = 2. Concentrations are mean values of the triplicate ± standard deviation; n.a. = not analyzed, < LOQ = below limit of quantification.

	Technosols						Soil	CDW mixtures	
	<i>a75</i>	<i>a50</i>	<i>a25</i>	<i>b75</i>	<i>b50</i>	<i>b25</i>	<i>ctl</i>	RCM1	RCM2
pH [-]	8.7 ±0.1	8.8 ±0.0	8.7 ±0.0	8.8 ±0.1	8.7 ±0.1	8.7 ±0.0	8.5 ±0.0	10.5 ±0.5	10.3 ±0.3
EC [µS/cm]	278 ±17.6	215 ±9.5	205 ±5.4	269 ±39.3	217 ±19.1	206 ±4.0	212 ±2.9	552 ±28.1	503 ±65.3
Fluoride [mg/L]	0.6 ±0.2	0.5 ±0.0	0.5 ±0.0	0.4 ±0.0	0.5 ±0.0	0.5 ±0.0	0.5 ±0.0	0.6 ±0.0	0.6 ±0.1
Chloride [mg/L]	1.6 ±0.9	< LOQ	< LOQ	< LOQ	2.0 ±0.8	2.2 ±0.9	< LOQ	3.7 ±1.9	1.4 ±0.6
SO <sub>4</sub> [mg/L]	52.3 ±6.0	26.7 ±2.4	11.2 ±1.4	44.0 ±10.4	19.0 ±0.8	9.6 ±1.4	1.4 ±0.5	167 ±41.9	170 ±8.2
Sb [µg/L]	< LOQ	3.3 ±1.2	4.2 ±1.2	3.3 ±1.2	< LOQ	< LOQ	3.3 ±1.2	< LOQ	< LOQ
As [µg/L]	8.3 ±0.5	7.0 ±0.8	< LOQ	4.8 ±1.6	5.7 ±0.9	< LOQ	< LOQ	< LOQ	< LOQ
Pb [µg/L]	< LOQ	8.0 ±2.2	4.3 ±2.6	3.3 ±1.2	4.3 ±2.6	5.8 ±2.7	< LOQ	< LOQ	< LOQ
B [µg/L]	150 ±8.2	96.7 ±17.0	70.0 ±0.0	100 ±8.2	103 ±26.2	73.3 ±4.7	< LOQ	193 ±17.0	143 ±60.2
Cd [µg/L]	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ
Cr [µg/L]	< LOQ	4.3 ±2.6	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	23.3 ±2.5	37.3 ±10.8
Cr(VI) [µg/L]	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	25.0 ±2.9	38.7 ±10.9
Co [µg/L]	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ
Cu [µg/L]	< LOQ	3.3 ±1.2	3.3 ±1.2	< LOQ	< LOQ	3.3 ±1.2	< LOQ	< LOQ	< LOQ

Mo [µg/L]	8.0 ±0.8	5.3 ±0.5	6.0 ±0.0	6.3 ±0.5	5.3 ±0.5	4.8 ±1.6	4.5 ±1.5	9.8 ±5.3	2.5 ±0.0
Ni [µg/L]	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ
Hg [µg/L]	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ
Se [µg/L]	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ
Tl [µg/L]	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ
V [µg/L]	43.3 ±4.7	33.3 ±4.7	16.7 ±4.7	26.7 ±4.7	23.3 ±4.7	20.0 ±0.0	5.0 ±0.0	166.7 ±47.1	66.7 ±9.4
Zn [µg/L]	< LOQ	25.0 ±0.0	25.0 ±0.0	25.0 ±0.0	25.0 ±0.0	25.0 ±0.0	25.0 ±0.0	25.0 ±0.0	25.0 ±0.0
Sn [µg/L]	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ
DOC [mg/L]	5.7 ±0.5	6.3 ±0.5	6.7 ±0.5	5.7 ±0.5	5.7 ±0.5	7.3 ±1.2	6.0 ±0.8	n.a.	n.a.

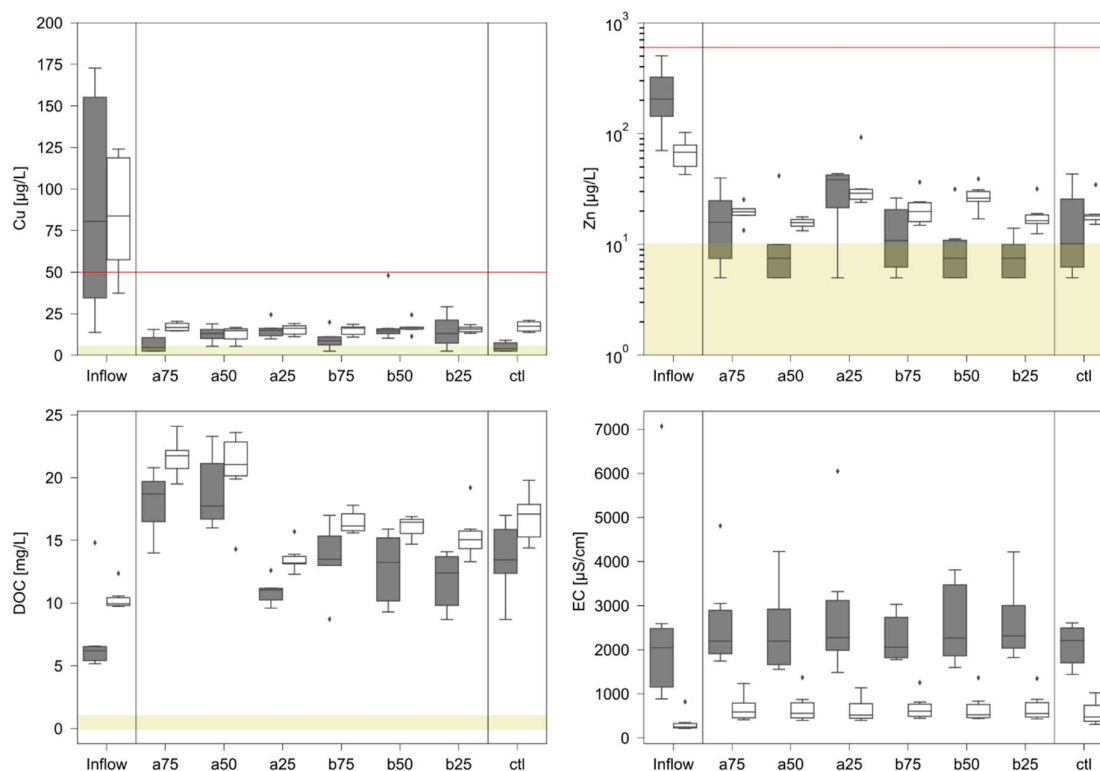
Most metal concentrations, also those for Cu and Zn, in the eluate of all CDW mixtures, the six technosols, and the control were low and under the threshold values of TV\_2.

Exceptions were V, and Cr(VI). Here, the threshold values for TV\_2 were exceeded. The highest V concentrations in the eluate were found for the CDW mixtures between 66.7 µg/L (RCM2) and 167 µg/L (RCM1), slightly lower concentrations for the six technosols (up to 43.3 µg/L for *a75*). Also, the V concentrations of 5 µg/L in *ctl* were higher than the threshold values of 4 µg/L in TV\_2. The Cr(VI) concentrations were highest in the CDW mixtures with 25.0 µg/L (RCM 1) and 38.7 µg/L (RCM 2). In comparison, the threshold value TV\_2 is 8 µg/L. The technosols and the *ctl* did not exceed the threshold value for Cr(VI).

Sulfate concentrations in the eluate of the two CDW mixtures RCM1 and RCM2 were the highest. However, in all technosols, sulfate concentrations were lower, ranging from 9.6 mg/L (*b25*) to 52.3 mg/L (*a75*). Fluoride concentrations in the eluate of all tested technosols, RCM1 and RCM2, and *ctl* were low and did not exceed the threshold values of TV\_2 which is 1.5 mg/L. Additionally, chloride concentrations were also low. DOC concentrations in the technosols and *ctl* were low and ranged from 5.7 to 7.3 mg/L.

### 3.2. Retention and Water Quality

Figure 2 shows the retention performance for Cu and Zn from the traffic area runoff by the different technosols compared to *ctl* (cf. Table A3).



**Figure 2.** Cu, Zn, DOC and EC; grey boxes = RI\_1, white boxes = RI\_2; red solid line = TV\_1, yellow areas = limits of quantification.

Mean influent concentrations of Cu were 73.4 µg/L at RI\_1 and 84.7 µg/L at RI\_2. While the Cu concentrations in the influent of the lysimeters were still above the threshold values of TV\_1 (50 µg/L), in the leachate of all experiments (RI\_1 and RI\_2), the values are consistently below 20 µg/L. However, we also analyzed the seepage water between June 2023 and February 2024 for mean background concentrations, the background values (5 to 6.5 µg/L) were lower compared to the leachate concentrations (up to 19.4 µg/L for *a50*) after treatment with traffic area runoff. Based on Mann-Whitney U tests with a significance level of  $\alpha=0.05$ , the effluents of *a50*, *a25*, and *b50* at RI\_1 showed significantly higher Cu concentrations than the control ( $p=0.019$ , 0.005, and 0.005, respectively). At RI\_2, the effluents of the technosols showed no significant differences in Cu concentrations compared to the control.

Similar observations can be made for zinc, but differences between RI\_1 and RI\_2 (Figure 2). Mean influent concentrations of Zn were 247 µg/L at RI\_1 and 68.1 µg/L at RI\_2, both below the threshold value TV\_1 of 600 µg/L. Mean effluent concentrations of Zn of all technosols and the control were much lower, ranging from 9.4 µg/L (*a25*) to 16.2 µg/L (*a50*) at RI\_1 and 15.7 µg/L (*a50*) to 38.5 µg/L (*a25*) at RI\_2. RI\_2 concentrations were higher compared to RI\_1 and higher than the background values. Based on Mann-Whitney U tests with a significance level of  $\alpha=0.05$ , effluent concentrations of Zn of all technosols including the control were significantly lower than in the influent (cf. Table A2) both at RI\_1 and RI\_2. The Zn discharges showed no significant differences compared to the control at RI\_1. At RI\_2, *a25* showed marginally higher Zn concentrations in the effluent compared to the control ( $p = 0.041$ ).

Mean influent concentrations of DOC were 7.4 mg/L at RI\_1 and 10.4 mg/L at RI\_2. Mean effluent concentrations of DOC were in the range of 9.4 (*a25*) to 15.4 mg/L (*a75*) at RI\_1 and 13.6 (*a25*) to 21.6 mg/L (*a75*) at RI\_2. DOC was significantly higher in the effluents of all technosols and the control compared to influent concentrations at RI\_1 and RI\_2. Compared to the control, effluent DOC was

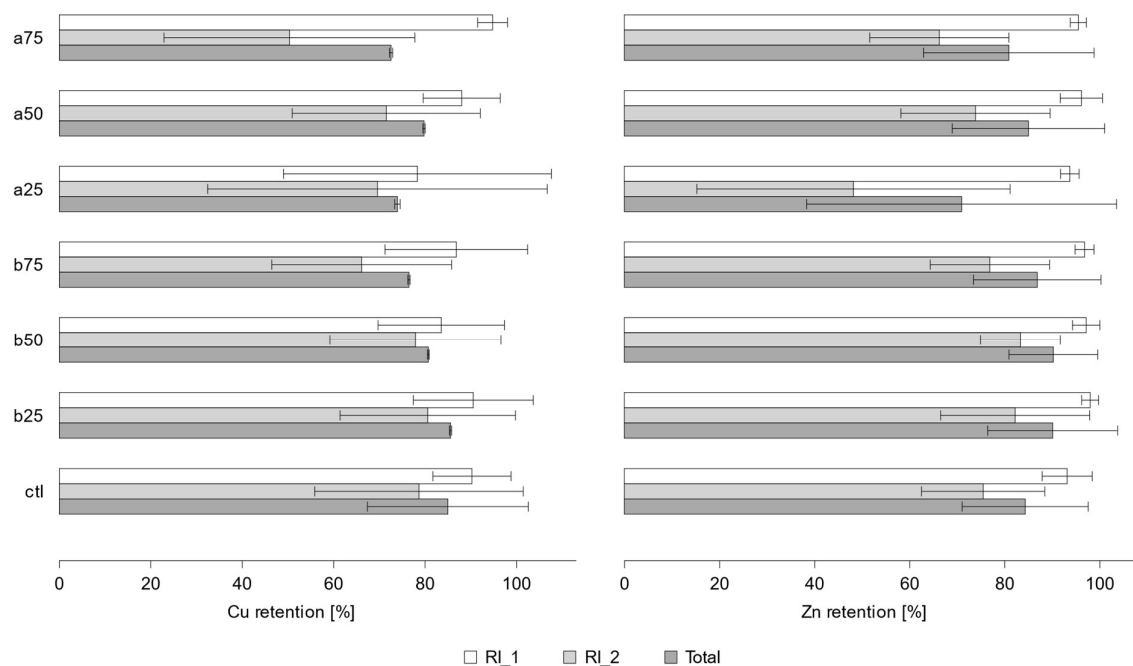


significantly higher in *a75* and *a50* at RI\_1. At RI\_2, effluent DOC was significantly higher in *a75* and significantly lower in *a25* compared to the control.

The pH values were comparable stable and were between 7.5 and 7.9 for all tests and technosols and the *ctl*.

The mean influent EC was 2,588  $\mu\text{S}/\text{cm}$  at RI\_1 and 352  $\mu\text{S}/\text{cm}$  at RI\_2. Mean effluent EC ranged from 2,270  $\mu\text{S}/\text{cm}$  (*b75*) to 2896  $\mu\text{S}/\text{cm}$  (*a25*) at RI\_1 and from 643  $\mu\text{S}/\text{cm}$  (*a25*) to 703  $\mu\text{S}/\text{cm}$  (*b25*) (*ctl* 576.5  $\pm$  257.4) at RI\_2. At RI\_1, the EC of effluents of all technosols, including the control, were not significantly different from influent EC. At RI\_2, the EC of effluents of all technosols, including the control, was, on the other hand, significantly higher than the influent EC. Compared to the control, no technosol effluent showed significant differences in EC at RI\_1 and RI\_2.

We evaluated the retention performance of the technosols for Cu and Zn for the tests depending on the rainfall intensities (Figure 3) based on the total loads (cf. Table A4). At RI\_1, mean retention efficiencies for Cu ranged from 78.3 % (*a25*) to 94.8 % (*a75*) (*ctl*: 90.2 %) and from 50.3 % (*a75*) to 80.6 % (*ctl* 78.7 %) at RI\_2. At RI\_1, mean retention efficiencies for Zn ranged from 93.7 % (*a25*) to 98.0 % (*b25*) (*ctl*: 93.2 %) and from 48.2 % (*a25*) to 83.3 % (*b50*) (*ctl* 75.5 %) at RI\_2. Overall mean retention efficiencies of Cu were highest in *b25* (85.6 %) and lowest in *a75* with 72.5 % (*ctl* 73.9 %). Overall mean retention efficiencies of Zn were highest in *b50* with 90.3 % and lowest in *a25* with 71.0 % (*ctl* 84.3 %).

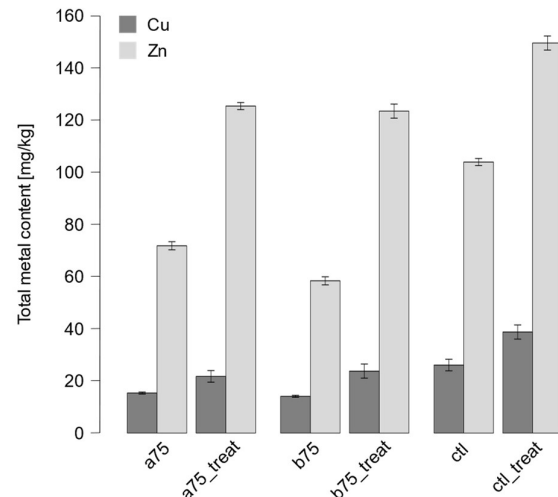


**Figure 3.** Retention efficiencies of Cu and Zn based on influent and effluent loads for RI\_1, RI\_2, and total (= both rain intensities).

RI\_1 and RI\_2, showed no significant differences in Cu retention of technosols compared to the control soil. At RI\_1, there were no significant differences in Zn retention of technosols compared to the control. At RI\_2, only *a25* showed significantly lower Zn retention efficiency than the control ( $p = 0.03$ ). Total retention efficiencies of technosols showed no significant differences compared to the control. However, across all soils, retention efficiencies for Cu and Zn were significantly reduced at higher rainfall intensity ( $p = 0.003$  and  $p < 0.001$ , respectively).

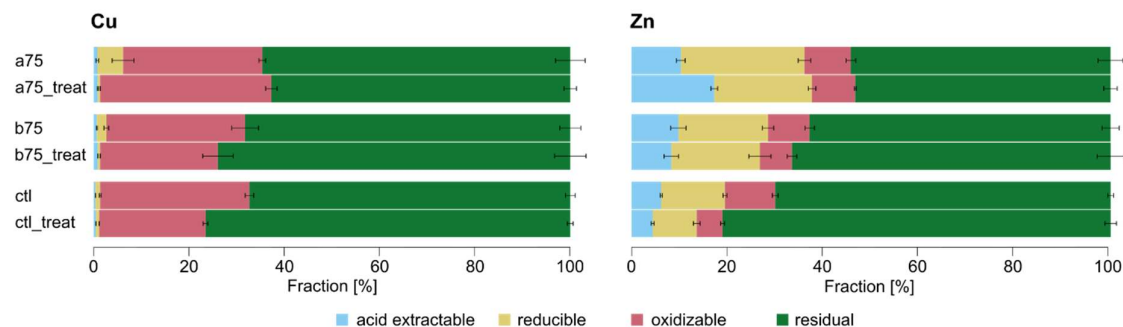
### 3.3. Sequential Extraction of Cu and Zn

After treatment with TAR, Cu and Zn accumulation was measured in the technosols *a75* and *b75* and the control (Figure 4).



**Figure 4.** Total contents of Cu and Zn in the technosols *a75*, *b75* and *ctl* before treatment and the technosols after treatment with TAR (*a75\_treat*, *b75\_treat* and *ctl\_treat*), based on sums of S1-4 derived from the sequential extraction procedure.

Based on the sequential extraction, the residual Cu fraction was the dominant component in all samples and always accounted for more than 60% of the total content (Figure 5). In the untreated substrates, the residual Cu content is higher in *ctl* compared to *a75* and *b75*. The following order was determined in the treated soils: *ctl\_treat* > *b75\_treat* > *a75\_treat*.



**Figure 5.** Fractions of mobilizable (acid extractable, reducible, and oxidizable) and residual phases before and after treatment with TAR based on the sequential extraction procedure.

The second largest Cu fraction in all substrates was the oxidizable, accounting for approximately 30% in the untreated soils, 22.3 % in *ctl\_treat* and 35.9 % in *a75\_treat*. Again, in the untreated substrates, the Cu contents were higher in *ctl* compared to *a75* and *b75*. A similar pattern was seen in the treated substrates: *ctl\_treat* > *a75\_treat* > *b75\_treat*.

The reducible and acid-exchangeable Cu fractions were < 1 mg/kg in all samples, and each represented less than 2% of the total contents. An exception was *a75*, where 0.87 mg/kg was measured for the acid exchangeable Cu fraction, corresponding to 5.45 %.

Considering the relative frequencies of mobilizable Cu (sum of S1-S3), *a75\_treat* and *b75\_treat* showed increased mobile phases of Cu compared to *ctl\_treat*.

In summary, the proportion of the residual Cu fraction increased after treatment while the mobile Cu phases (S1-S3) decreased, except for *a75\_treat*. The mobile Cu phase increased slightly, shifting from reducible to oxidizable fractions.

Of all treated soils, *ctl\_treat* had the highest total Zn content, followed by *a75\_treat* and *b75\_treat*. However, the content of Zn in the untreated substrates was also higher in *ctl* than in *a75* and *b75*. In the untreated soils, the fractionation obtained from BCR-SEP for *a75* and *b75* was as follows: residual >> reducible >> acid exchangeable > oxidizable, while for *ctl*, the order was: residual >> reducible > oxidizable > acid exchangeable. In the treated substrates, the fractionation in *ctl\_treat* was identical to *ctl*, while in *a75\_treat* and *b75\_treat*, it was: residual >> reducible > acid exchangeable > oxidizable.

The residual Zn fraction was dominant in all samples. The reducible Zn fraction was the second largest in all samples. The highest reducible Zn fraction was found in substrate *a75*, followed by *ctl* and *b75*. The treated samples showed the following order: *a75\_treat* > *b75\_treat* > *ctl\_treat*. The substrates *a75* and *b75* differ from *ctl* in terms of Zn fractionation. In the control soil, the oxidizable Zn fraction ranked third in the untreated and treated. In *a75* and *b75*, the acid exchangeable Zn fraction was the third largest.

The lowest Zn contents of *ctl* were found in the acid exchangeable fraction, both treated and untreated. For *a75* and *b75*, the oxidizable Zn fraction was the smallest in treated and untreated. In summary, the residual fraction increases after TAR treatment while the mobile phase decreases, except in *a75\_treat*. There, the proportion of the mobile Zn phase remains nearly unchanged, with a shift of the reducible fraction towards the acid-exchangeable fraction.

Comparison of the soils after TAR treatment

To assess shifts in the chemical bindings of Cu and Zn after treatment with TAR, the treated substrates’ mean Cu and Zn contents were subtracted from those of the untreated soils (Table 4). The most pronounced shifts of Cu contents were found for *a75*, where oxidizable Cu was nearly as high as residual Cu, showing a decrease in reducible Cu on the other hand. *b75* also showed a relative increase in oxidizable Cu, albeit less pronounced. In *ctl*, most Cu accumulation occurred in the residual phase. Analogous, the most pronounced shifts of Zn were found in *a75*, where a significant accumulation of Zn was found in the acid exchangeable phase, and only half of the Zn accumulation was found in the residual phase. In *b75*, most of the Zn accumulation occurred in the residual phase. In *ctl*, all highly mobile phases stayed the same or decreased, and only the residual phase increased.

**Table 4.** Cu and Zn shifts in the substrates; calculated as: Mean percentage of Cu and Zn of treated soils minus mean percentage in untreated substrates [%].

Metal	Substrate	S1 Acid exchangeable	S2 Reducible	S3 Oxidizable	S4 Residue	Sum S1-S4
Cu	<i>a75</i>	1.1	-11.8	52.2	58.6	100
	<i>b75</i>	1.2	-1.7	17.2	83.3	100
	<i>ctl</i>	0.8	-0.1	3.9	95.5	100
Zn	<i>a75</i>	26.5	13.3	8.0	52.2	100
	<i>b75</i>	7.0	17.7	4.9	70.3	100
	<i>ctl</i>	0.2	-0.1	-6.2	106.1	100

4. Discussion

4.1. Risk of Substance Release

Except for Cr(VI) and V, the leaching of substances obtained from batch tests at L/S 2 was largely unremarkable compared to the German threshold values for soil solution TV\_2. Concentrations of Cr(VI) in both RCM1 and RCM2 exceeded the threshold value substantially. However, Cr(VI) concentrations were below the threshold value in the six technosols, which will be normally applied in practice. The concentration of V in RCM1 exceeded the threshold value TV\_2 and was substantially higher than in RCM2. Hence, while V concentrations were lowest in the control, in the technosols, we see a dose-response of V with an increasing ratio of RCM1, which, in contrast, is vaguer for RCM2.

Crushed bricks release substantially more sulfate, Cr, and V than crushed concrete [24]. Vanadium release from CDW that contains bricks can be three times higher than in CDW based solely on concrete [13]. Our data confirm that an increased proportion of bricks in the CDW increases the release of some elements like Cr and V [13,24–26]. While in German legislation, an L/S-dependent decay behavior is assumed to evaluate recycled building materials for Cr discharges, there is no generalizable decay behavior for V [27]. While the high pH of mixed CDW is related more to its concrete than its brick phase [24], in our study, the pH of both CDW mixtures derived from the batch tests was high ( $> 10$ , cf. Table 3). Over time, the increased specific surface area and increased air and water supply during storage facilitate the carbonation of crushed CDW [13], resulting in a pH between 8 and 9 [28–31] while partly carbonated CDW is suggested to have a pH 10.5 to 11.9 [29,32]. This indicates that the CDW mixtures were not yet carbonated as they were sampled. Carbonation is associated with increased leaching of anionic compounds when the aging of concrete leads to the release of sulfate and its substituting ions, chromate, and vanadate [13,33]. Consequently, in our study, the sulfate leaching from the mixed CDW, the technosols, and the control correlated strongly with the leaching of Cr(VI) ( $r^2 = 0.81$ ) and V ( $r^2 = 0.71$ ). Furthermore, V release in soils is supposed to correlate to higher pH [34]. Therefore, we attribute the reduced release of V from the soil to a reduction in the pH value by adding soils with a neutral pH and to a reduced release of sulfate in the technosol mixtures. For EC, we observed a dose-dependent response, increasing with higher ratios of both RCM 1 and RCM 2.

#### 4.2. Retention and Binding

The traffic area runoff TAR concentrations of Cu and Zn used as influent in this study were comparable with concentrations reported for urban roads with AADT  $> 15,000$  vehicles [1]. The Zn/Cu ratio at RI\_1 was 3.4, which concurs with the literature [1]. However, Zn concentrations and thus Zn/Cu ratio of TAR used for RI\_2 (0.8) were lower than expected. We attribute this to the different sampling periods. The TAR for RI\_1 was sampled in late winter/early spring, whereas the TAR for RI\_2 was sampled during the summer months. In winter, metal loadings in traffic area runoff are expected to be higher, which is often caused by the application of de-icing salts and increased corrosion rates [1,35,36].

Metals from TAR were found to bind to soil particles by precipitation or adsorption, forming strong bonds that are difficult to change under typical environmental conditions [1,37]. Copper occurs in topsoils in oxic conditions, mostly in oxidation state II, and is not very exchangeable at pH  $> 5$ ; its solution concentration is mainly determined by adsorption and desorption, depending on pH and soluble, organic or inorganic complexing agents [38]. We assume that due to the high carbonate content of the soils analyzed, Cu is present in the form of carbonate and organometallic complexes [38]. In addition, we assume that the microbial degradation of the substrate compost releases soluble organic complexing agents and promotes the mobilization of adsorbed Cu [38]. On the other hand, the content of exchangeable Zn is very low at pH  $> 6.5$  as  $\text{Zn}^{2+}$ ,  $\text{Zn}(\text{OH})^+$ , and  $\text{ZnCO}_3$  are mainly present here, of which  $\text{Zn}(\text{OH})^+$  in particular increases proportionally with increasing pH and is specifically adsorbed and fixed by oxides [38]. Furthermore, metals transported by the TAR to roadside soils are bound to particles, which in turn are largely retained by physical mechanisms, particularly in the upper soil layers [2,39]. Therefore, metal mobilities were determined in roadside soils depending on pH and organic matter [40–42].

Accordingly, Zn retention in our study was higher than Cu retention, comparable to findings reported from the US [43]. Our study found no correlations between Cu and Zn discharge in the effluents and its pH, EC, or DOC. Although DOC concentrations in the effluents were higher than in the influents (presumably due to the addition of compost), we suggest that due to the high charging rates applied in our study, retention times in the soil were not long enough to promote the complexation of Cu and organic matter. In addition, we can rule out unfavourable redox conditions due to the high oxygen content in the effluents. As the Cu and Zn retentions measured for RI\_2 were significantly lower than RI\_1, we consider it rather likely that the mechanical filtering effect of the

coarse-grained soils with high water permeability was no longer present at the high charging rate. Hence, we assume that retention of particulate-bound Cu and Zn was impaired. This assumption is supported by the fact that there was no backwater in the plots during the irrigations, even at a high charging rate. Nevertheless, the Cu and Zn retentions of the studied technosols were not significantly lower than that of the control. The effluent concentrations of Cu and Zn of all analyzed technosols and the control were below the threshold of the German BBodSchV.

After the treatment with TAR, the direct AR pseudo-total analysis was generally unable to extract the same metal content as the sum of the BCR-SEP fractions (S1-4). However, we found accumulations compared to the non-treated soils for Cu and Zn. The relatively lower concentrations in the pseudo-total can also be reflected in the pseudo-accuracy of the reference material BCR-701, with the residual fraction slightly overestimated and the direct AR pseudo-total rather underestimated (cf. Table A5). The large difference cannot be fully explained. Possible reasons could be that the samples were not ground before direct digestion, leading to incomplete extraction of metal concentrations and that the AR digestion technique is more accurate for analyzing the residue than the raw material [19]. Davidson, et al. [44] and Rommel, Stinshoff and Helmreich [19] reported comparable deviations of summarized SEP fractions and pseudo-total digestion from sequential extractions of soil and filter media, respectively. Also, for these reasons, the recoveries are sometimes very poor (cf. Table A5). They are in the 109-234% range, which means that the sum of the fractions of metal concentrations is sometimes more than twice as high as the pseudo-total. Ideally, the values should be between 80-120% [45].

For Cu and Zn, the residual fraction increases slightly after treatment (approx. 2-11%), except in *a75\_treat*, where a small increase in the mobile phase was observed. The residual fraction is >53% in all substrates. Due to the pseudo-accuracy calculated for this fraction, we attribute discrepancies to overestimating the residual fraction. Sutherland, et al. [46] reported lower residual fractions of Cu (35 %) and Zn (21%) in road-deposited sediments (RDS). In contrast, Pérez, López-Mesas and Valiente [37] found residual fractions of up to 80% for Cu in RDS. Rommel, Stinshoff and Helmreich [19] have found comparably high residual fractions in carbonate sand used to treat TAR from the same origin we used in this study. Zn and Cu are found predominantly in particulate form in traffic area runoff TAR, making the upper soil layer critically important. In this layer, metals are primarily retained mechanically by sedimentation and filtration, while chemical processes such as adsorption and binding to organic matter also contribute significantly to their immobilization [1,2]. Rommel, Stinshoff and Helmreich [19] sampled the TAR used in this study at the same sampling site as in this study and found concentrations of total Cu and Zn approx. 7 times higher than dissolved Cu and Zn.

For Cu, the oxidizable fraction is the second largest (approx. 22 to 36 %), while for Zn, it is negligible (about 5 to 11 %). In *a75\_treat*, treatment with TAR caused a shift of Cu from the reducible fraction (from 5.5 to 0.45 %) to the oxidizable fraction (from 29.2 to 35.9 %). Bacon and Davidson [47] point out that Cu expected in the oxidizable can also be released in the reducible phase, making interpretation difficult. Similar contents in the oxidizable fraction (approximately 26 %), which also correlate with organic carbon, were observed by Sutherland, et al. [48] and Kartal, et al. [49] in studies of RDS in Honolulu, Hawaii, and Kayseri, Turkey (cf. Table A6).

*a75\_treat* also shows a shift of Zn, with a decrease in the reducible fraction (from 25.8 to 20.4 %) and an increase in the acid-exchangeable fraction (from 10.3 to 17.3 %). The latter, the most mobile and potentially harmful fraction, is strongly influenced by pH. Metals become more mobile the more acidic the soil. This behaviour is opposite to the pH in the substrates, especially for *a75\_treat* and *a75\_treat*. Bacon and Davidson [47] also found that a phase shift can occur during the extraction of Zn, which can distort the results. The findings of this study differ from other studies, where higher fractions of acid-exchangeable Zn were reported, ranging from 25 to 38 % [37,46,48,49].

A comparison of only the mobile fraction (S1-S3) shows a similar pattern in fractionation for Zn in the substrates *a75\_treat* and *b75\_treat* as in other studies [46,48,49]. However, the proportions of the individual fractions vary considerably, with overall higher Cu and Zn contents, especially in the acid-exchangeable fraction reported in these comparative studies.



No comparable pattern emerges for Cu. However, [37] reported a similar sequence of fractions, with the residue even accounting for about 80 %. In contrast, Kartal, Aydın and Tokaloğlu [49] and Sutherland, Tack and Ziegler [46] reported the following order for Cu: acid-exchangeable < oxidizable < reducible. In contrast to *b75\_treat* and *ctl\_treat*, in *a75\_treat* was an increase of Cu in both the mobile and residual phase.

*b75*, which contains higher proportions of concrete, mortar, and plaster, shows favourable distribution ratios than *a75*. This finding is in concordance with Pallewatta, et al. [50], who have found better retention in concrete-based waste than in masonry-based waste. Barrett, Katz and Taylor [3] point out that Portland cement concrete in roadside soils fosters metal retention mainly by controlling pH and precipitation. Furthermore, we hypothesize that in *a75*, where higher iron contents due to a higher brick ratio are to be expected, the presence of humic-coated Fe-oxide colloids combined with also higher Ca contents inherent in the CDW promotes the transport of Cu [51]. On the other hand, high Cu retention [6,7] and Zn retention [52] were found for brick or brick-dominated aggregates.

However, despite overall comparable Cu and Zn retentions when charged with TAR, both CDW-amended technosols showed unfavourable binding properties compared to the control. This is due to relative increases of mobile Cu and Zn phases which could be released by various environmental influences (e.g., changes in pH, de-icing salt input, enrichment of organic matter).

## 5. Conclusions

Technosols amended with brick-dominated CDW showed an dose-dependent increased leaching potential of V, which correlates with sulfate emissions. Therefore, V and sulfate content should always be analyzed and high contents should be avoided when bricks are used to amend topsoils of infiltration swales to not harm groundwater quality. However, CDW-amended technosols can be used in infiltration swales regarding the retention of metals from traffic area runoff. They had comparable Cu and Zn retentions as the control soil with the same particle size distribution. Considering the chemical binding of Cu and Zn, the amendment of concrete-dominated CDW is favourable to brick-dominated CDW. Still, both showed an increase of mobile Cu and Zn phases compared to the control, implying an increased potential for desorption and remobilization. Considering sustainability, using recycled construction and demolition waste in infiltration swales could be a sustainable solution for reducing CO<sub>2</sub> emissions and resource consumption.

**Author Contributions:** SK: Conceptualization; Data curation; Formal analysis; Funding acquisition; Investigation; Methodology; Project administration; Resources; Software; Supervision; Validation; Visualization; Writing — original draft. JM: Investigation; Writing — review and editing. PS: Writing — review and editing. BH: Supervision; Writing — review and editing. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research was funded by the City of Munich, Municipal Department and contributes to the EU URBACT project URGE (circUlaR buildinG citiEs). Parts of this work were additionally funded by The German Research Foundation (DFG) funds the Research Training Group Urban Green Infrastructure – Training Next Generation of Professionals for Integrated Urban Planning Research (GRK 2679/1).

**Data Availability Statement:** The data presented in this study are available on request from the corresponding author due to legal restrictions from the funding partner.

**Acknowledgments:** The authors thank Myriam Reif, Wolfgang Schröder, Dr. Carolin Heim, and Maximilian Damberger from the Technical University of Munich for their technical and analytical support. The authors thank the City of Munich's Municipal Department for funding this study.

**Conflicts of Interest:** The authors declare no conflicts of interest. The funders had no role in the study's design, in the collection, analyses, or interpretation of data, in the writing of the manuscript, or in the decision to publish the results.

## Abbreviations

The following abbreviations are used in this manuscript:

AR	Aqua regia
BCR	Community Bureau of Reference, European Commission
CDW	Construction and demolition waste
RCM	Recycling mixture (in this study: crushed aggregates of mixed CDW)
RDS	Road-deposited sediments
SEP	Sequential extraction procedure
TAR	Traffic area runoff
TV_1	Threshold values for seepage water according to BBodSchV
TV_2	Threshold values for soil solution according to BBodSchV

Appendix A

Table A1. List of methods, limits of quantification (LOQ), and threshold values according to BBodSchV.

Substance		Method	LOQ	Unit	BBodSchV [15]	
					TV_1	TV_2
Batch tests						
pH		DIN 38404-5: 2009-07				
EC		DIN EN 27888: 1993-11	10	µS/cm		
Sb	AR	DIN EN ISO 17294-2: 2017-01	5	µg/L	5	10
As	AR	DIN EN ISO 17294-2: 2017-01	5	µg/L	10	25
Pb	AR	DIN EN ISO 17294-2: 2017-01	5	µg/L	10	85
B	AR	DIN EN ISO 17294-2: 2017-01	50	µg/L	1000	1000
Cd	AR	DIN EN ISO 17294-2: 2017-01	0.5	µg/L	3	7,5
Cr	AR	DIN EN ISO 17294-2: 2017-01	5	µg/L	50	50
Cr(VI)	AR	DIN ISO 15923-1: 2014-07	5	µg/L	8	8
Co	AR	DIN EN ISO 17294-2: 2017-01	5	µg/L	10	125
Cu	AR	DIN EN ISO 17294-2: 2017-01	5	µg/L	50	80
Mo	AR	DIN EN ISO 17294-2: 2017-01	5	µg/L	35	70
Ni	AR	DIN EN ISO 17294-2: 2017-01	5	µg/L	20	60
Hg	AR	DIN EN ISO 12846: 2012-08	0.2	µg/L	1	1
Se	AR	DIN EN ISO 17294-2: 2017-01	5	µg/L	10	10
Tl	AR	DIN EN ISO 17294-2: 2017-01	0.5	µg/L		
V	AR	DIN EN ISO 17294-2: 2017-01	2	µg/L	4	70
Zn	AR	DIN EN ISO 17294-2: 2017-01	50	µg/L	600	600
Sn	AR	DIN EN ISO 17294-2: 2017-01	20	µg/L		
DOC	Filtration 0.45 µm	DIN 1484	1	mg/l		
Cl		DIN ISO 15923-1: 2014-07	2	mg/l		
SO <sub>4</sub>		DIN ISO 15923-1: 2014-07	2	mg/l		
F		DIN 38405-4: 1985-07	0.2	mg/l	1,5	
Retention tests						
pH		on-site				
EC		on-site		µS/cm		
O <sub>2</sub>		on-site		mg/L		
Cu	AR	DIN EN ISO 17294-2: 2017-01		µg/L	50	
Zn	AR	DIN EN ISO 17294-2: 2017-01		µg/L	600	

Table A2. Mann-Whitney U statistics and p-values for Cu and Zn concentrations in the effluent of the technosols compared to the control.

Element	Rain intensity	Technosol	U Statistic	p-value
Cu	121 L/(s·ha)	a75	21	0.732
		a50	33	0.019
		a25	36	0.005
		b75	28	0.142

Zn	221 L/(s·ha)	b50	36	0.005
		b25	30	0.073
		a75	18	1.000
		a50	8	0.132
		a25	12	0.394
		b75	11	0.310
		b50	15	0.699
		b25	11.5	0.336
	121 L/(s·ha)	a75	19	0.935
		a50	13	0.452
		a25	26.5	0.196
		b75	16.5	0.870
		b50	14.5	0.618
		b25	12	0.357
	221 L/(s·ha)	a75	21.5	0.630
		a50	6.5	0.078
		a25	31	0.041
		b75	19	0.937
		b50	28	0.132
		b25	14	0.589

**Table A3.** Mean background concentrations and mean influent and effluent concentrations from TAR treatment; L/S = liquid to solid ratio at the beginning of TAR treatment; inf = inflow; n.a. = not analyzed.

	Mean background concentrations (n = 4)				Concentrations rain intensity RI_1 (n = 6)					concentrations rain intensity RI_2 (n = 6)				
	L/S	Cu [µg/L]	Zn [µg/L]	DO C [mg/L]	Cu [µg/L]	Zn [µg/L]	DO C [mg/L]	p H	EC [µS/cm]	Cu [µg/L]	Zn [µg/L]	DO C [mg/L]	p H	EC [µS/cm]
inf	n.a.	n.a.	n.a.	n.a.	73.4 ±61.5	247 ±169	7.4 ±3.9	7.8 ±0.1	2,588 ±2,128	84.7 ±34.6	68.1 ±20.5	10.4 ±0.9	7.8 ±0.1	351 ±218
a75	0.51	6.5 ±2.6	6.5 ±2.6	35 ±14	7.0 ±5.1	17.5 ±13.8	15.4 ±6.7	7.7 ±0.1	2,648 ±1060	17.1 ±2.4	19.6 ±3.6	21.6 ±1.5	7.8 ±0.1	683 ±286
a50	0.57	7.0 ±3,5	5 ±0	31 ±13	12.7 ±4.4	14.6 ±14.3	16.2 ±7.1	7.9 ±0.3	2,471 ±948	12.7 ±4.3	15.7 ±1.5	200.6 ±3.1	7.8 ±0.1	697 ±3389
a25	0.55	6.5 ±2.6	5 ±0	38 ±18	15.3 ±4.7	24.8 ±18.8	9.4 ±3.9	7.5 ±0.1	2,896 ±1520	15.4 ±3.1	38.5 ±24.3	13.6 ±1.1	7.9 ±0.0	643 ±268
b75	0.47	5 ±0	5 ±0	23 ±10	9.5 ±5.5	11.6 ±9.1	11.6 ±5.3	7.7 ±0.1	2,270 ±516	15.1 ±2.9	21.8 ±7.4	16.5 ±0.8	7.8 ±0.0	698 ±277

b50	0.57	5 ±0	5 ±0	20 ±7	19.4 ±12.9	9.7 ±9.5	11.0 ±5.1	7.9 ±0.3	2,590 ±901	16.9 ±3.8	27.3 ±6.7	16.1 ±0.8	7.8 ±0.1	688 ±330
					14.5 ±9.3	8.3 ±4.8	10.1 ±4.6	7.8 ±0.1	2,643 ±829	15.7 ±1.8	18.6 ±6.2	15.5 ±1.9	7.7 ±0.1	703 ±323
					5.0 ±2.8	14.1 ±15.1	11.6 ±5.4	7.9 ±0.2	2,099 ±453	17.4 ±2.9	20.2 ±6.6	16.9 ±1.9	7.7 ±0.0	576 ±257

Table A4. Total loads [mg] and load based retention [%] of Cu and Zn per substrate and rain intensity RI.

		Cu			Zn		
		Influent load	Effluent load	Retention	Influent load	Effluent load	Retention
a75	RI_1	101	2.8	94.8 ±3.3	158	7.4	95.5 ±1.7
	RI_2	27.4	7.5	50.3 ±27.4	27.2	8.4	66.3 ±14.6
	Total	128	10.3	72.5 ±0.4	185	15.8	80.9 ±18.0
a50	RI_1	110	6.1	88.0 ±8.4	225	5.6	96.2 ±4.4
	RI_2	46.0	6.0	71.5 ±20.6	40.2	7.3	73.9 ±15.7
	Total	156	12.0	79.8 ±0.3	265	12.9	85.0 ±16.0
a25	RI_1	146	6.0	78.3 ±29.3	191	12.7	93.7 ±2.0
	RI_2	131	6.2	69.6 ±37.1	40.4	14.6	48.2 ±32.9
	Total	276	12.2	73.9 ±0.6	232	27.3	71.0 ±32.6
b75	RI_1	80.9	4.0	86.8 ±15.6	172	5.3	96.8 ±2.0
	RI_2	31.5	5.9	66.1 ±19.7	39.4	8.5	76.9 ±12.6
	Total	112	9.9	76.5 ±0.3	211	13.8	86.9 ±13.4
b50	RI_1	111	8.5	83.5 ±13.8	2134	4.5	97.2 ±2.9
	RI_2	54.5	6.7	77.9 ±18.7	77.8	10.6	83.3 ±8.4
	Total	166	15.2	80.7 ±0.2	292	15.1	90.3 ±9.3
b25	RI_1	130	5.1	90.5 ±13.1	320	2.7	98.0 ±1.8
	RI_2	56.4	5.5	80.6 ±19.2	89.1	6.7	82.2 ±15.7
	Total	187	10.5	85.6 ±0.2	410	9.4	90.1 ±13.7
ctl	RI_1	75.7	2.1	90.2 ±8.5	150	7.3	93.2 ±5.3
	RI_2	105	7.3	78.7 ±22.8	48.9	9.1	75.5 ±13.0
	Total	181	9.4	73.9 ±17.6	199	16.3	84.3 ±13.3

Table A5. Cu and Zn content in S1 to S4 and their sum of the non-irrigated substrates a75, b75, and ctl and with TAR irrigated a75\_treat, b75\_treat, and ctl\_treat, reported as mean values ± standard deviation; additionally pseudo-total content and the recovery as a difference between sum S1-S4 and Pseudo-total.

Sample	S1 Acid exchangeable		S2 Reducible		S3 Oxidizable		S4 Residue		Sum S1-S4 <sup>a</sup>	Pseudo- total <sup>b</sup>	Recovery	
	[mg/kg]	[%]	[mg/kg]	[%]	[mg/kg]	[%]	[mg/kg]	[%]	[mg/kg]	[mg/kg]	[%]	
Cu	a75	0.13	0.82	0.87	5.45	4.66	29.2	10.3	64.5	16.1	8.28	193
		±0.06	±0.3	±0.48	±2.9	±0.26	±0.9	±0.42	±3.8	±0.69		
	b75	0.1	0.68	0.28	1.91	4.33	29.6	9.94	67.9	14.7	7.57	194
		±0.0	±0.1	±0.03	±0.6	±1.28	±3.4	±1.59	±2.7	±2.04		
	ctl	0.1	0.37	0.28	1.04	8.41	31.2	18.2	67.4	27.1	14.6	185
		±0.0	±0.0	±0.03	±0.2	±0.83	±1.1	±2.63	±1.3	±2.76		
a75_treat		0.2	0.89	0.1	0.45	8.05	35.9	14.1	62.8	22.5	17	132
		±0.0	±0.1	±0.0	±0.0	±0.34	±1.5	±1.57	±1.6	±1.61		

Zn	b75_treat	0.22	90	0.11	0.45	6.04	24.6	18.2	74.1	24.6	10.5	234
		±0.0	±0.1	±0.0	±0.0	±0.67	±4.0	±2.1	±4.0	±2.2		
	ctl_treat	0.2	0.5	0.27	0.67	8.93	22.3	30.7	76.6	40.1	18	223
		±0.0	±0.0	±0.02	±0.0	±0.49	±0.7	±2.81	±0.7	±2.91		
	a75	7.65	10.3	19.1	25.8	7.21	9.7	40.3	54.2	74.3	61.9	120
		±1.16	±1.0	±0.88	±1.6	±0.76	±1.3	±4.43	±3.1	±1.23		
	b75	5.73	9.49	11.4	18.9	5.37	8.89	37.9	62.8	60.4	49.6	122
		±0.50	±2.0	±3.08	±1.5	±1.79	±1.2	±8.7	±2.2	±4.33		
	ctl	6.66	6.2	14.3	13.3	11.2	10.5	75.3	70.1	107	79.6	135
		±0.18	±0.3	±1.3	±0.5	±0.39	±0.8	±5.54	±0.7	±12.4		
	a75_treat	22.4	17.3	26.5	20.4	11.6	8.98	69.2	53.3	131	108	120
		±1.16	±0.8	±0.62	±1.0	±0.07	±0.2	±4.09	±1.8	±7.18		
	b75_treat	10.4	8.17	23.3	18.3	8.68	6.8	85.3	66.8	128	60.1	213
		±1.27	±1.9	±2.20	±2.8	±2.59	±1.2	±15.9	±3.5	±4.32		
	ctl_treat	6.75	4.36	14.2	9.2	8.32	5.38	125	81.1	155	69.4	223
		±0.16	±0.3	±0.41	±0.8	±0.15	±0.4	±11.4	±1.5	±8.90		

<sup>a</sup> Sum of means of S1-S4, <sup>b</sup> Values from single determination

**Table A6.** Comparison of the sequentially extracted fractions of Cu and Zn in this study with data from the literature; RDS = Road desposited sediment or similiar.

Type		S1 Acid exchangeable [%]	S2 Reducible [%]	S3 oxidizable [%]	S4 residual [%]	total [mg/kg]
Cu						
a75	Technosol	0.8 ±0.3	5.4 ±2.3	29.2 ±0.7	64.6 ±3.1	15.9 ±0.4
a75_treat	Technosol	0.9 ±0.1	0.5 ±0.0	35.9 ±1.2	62.7 ±1.3	22.5 ±1.6
b75	Technosol	0.7 ±0.1	2.0 ±0.5	29.1 ±2.8	68.2 ±2.2	14.6 ±2.3
b75_treat	Technosol	0.9 ±0.0	0.5 ±0.0	24.7 ±3.2	73.9 ±3.3	24.6 ±1.4
ctl	Soil	0.4 ±0.0	1.0 ±0.2	31.3 ±0.9	67.3 ±1.0	27.0 ±2.8
ctl_treat	Soil	0.5 ±0.0	0.7 ±0.0	22.3 ±0.5	76.5 ±0.6	40.1 ±2.8
[46]	RDS	7.0 ±1.4	37.2 ±2.6	20.5 ±2.2	35.3 ±3.1	409.0
[49]	RDS	6.0	43.6	25.5	24.9	84.2
[37]	RDS	1.9	8.2	10.3	79.6	670.0
[48]	RDS	4.9 ±1.2	23.6 ±3.1	26.2 ±3.2	45.4 ±3.9	163.0
[53]	RDS	7.3	44.6	26.8	21.3	207.0
[19]	Carbonate sand	2.6 ±0.1	1.6 ±0.2	16.7 ±0.4	79.1 ±6.6	137 ±8
Zn						
a75	Technosol	10.3 ±0.9	25.8 ±1.3	9.7 ±1.0	54.2 ±2.6	74.3 ±4.2
a75_treat	Technosol	17.3 ±0.7	20.4±0.8	9.0 ±0.2	53.3 ±1.4	129.7 ±3.3
b75	Technosol	9.8 ±1.6	18.7 ±1.2	8.7 ±1.0	62.8 ±1.8	60.4 ±11.1
b75_treat	Technosol	8.3 ±1.5	18.5 ±2.3	6.7 ±1.0	66.5 ±2.8	128 ±15.0
ctl	Soil	6.2 ±0.2	13.3 ±0.4	10.5 ±0.6	70.0 ±0.6	107.5 ±5.6
ctl_treat	Soil	4.4 ±0.3	9.2 ±0.7	5.4 ±0.4	81.0 ±1.2	155 ±9.2
[46]	RDS	27.2 ±2.5	42.1 ±2.0	9.5 ±0.6	21.2 ±1.6	671
[49]	RDS	25.1	55.1	9.6	10.2	443
[37]	RDS	28.8	28.9	14.1	28.3	640
[48]	RDS	32.7 ±2.2	36.6 ±1.5	8.3 ±1.3	22.4 ±2.1	471
[53]	RDS	26.7	42.5	13.2	17.6	344
[54]	RDS	33.2	29.7	20.9	16.2	113.0
[19]	Carbonate sand	14.4 ±0.1	10.4 ±0.7	6.7 ±0.9	68.5 ±4.6	813 ±37

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