Preliminary analysis of soil samples from Squaw Creek National Wildlife Refuge, Missouri, for the dust suppressant EnviroKleen®

Project collaborator: USFWS

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Background

The health of soils along roadways is critical for maximizing habitat quality and minimizing negative ecological effects of roads. When dust suppressants or soil stabilizers are applied to roads, roadside soils may be exposed to these products through drift during initial applications, or by the movement of treated particulates by water or wind. Product residues in soils may then be available to plants, terrestrial invertebrates, or small mammals. However, very little work has addressed dust suppressant residues in soils.

As part of a larger, ongoing study on the environmental effects of dust suppressant chemicals on roadside plants and animals, we sampled roadside soils at Squaw Creek National Wildlife Refuge (NWR), Missouri. Road sections at Squaw Creek NWR had previously been treated with two road products, including EnviroKleen®. Because EnviroKleen is a synthetic iso-alkane, we predicted that it would have a distinctive signature in roadside soils that could be detected through gas chromatography/mass spectrometry (GC/MS).

Field test and application history

EnviroKleen® was applied to two 800-m sections of the Squaw Creek NWR auto tour loop on July 23-24, 2014, as part of a field test that also included applications of durablend-C™ and comparisons to an untreated control section. New aggregate had been placed on the road prior to product application. The untreated control section also received new aggregate, but no product. An application truck supplied by the product vendor, Midwest Industrial Supply, used a computerized spray bar system to apply EnviroKleen® at a rate of 1.36 L/m^2. Approximately 10 months later on May 12, 2015, the vendor completed an additional light "maintenance" application of EnviroKleen® on the same sections at a rate of 0.51 L/m^2. Therefore, product from the initial application weathered for ~11 months prior to soil sampling, whereas the product from the maintenance application weathered for ~2 months. Soil samples potentially contain residues from both these applications.

Figure 1. Layout of experimental treatment sections on the southern half of the Squaw Creek NWR auto tour loop. Each treated road section was 800 m. Refuge boundary in yellow.

Soil sampling

Soil samples were taken on June 17, 2015 along short transects placed perpendicular to the road, extending from georeferenced origination points at the road's edge within each treatment. Four perpendicular transects were plotted within each of five treated road sections (two EnviroKleen®-treated, two durablend-C™-

treated, and one untreated control), with sampling points at 1m and 4m from the road's edge (Figure 2). Only samples from EnviroKleen® and control treatments were analyzed for the presence of EnviroKleen®. Transects were placed on the east side of the road and in comparable habitats (vegetation, canopy cover, slope) to the greatest degree possible. Transects were concentrated in the middle of each road section to minimize influence from adjacent treated sections, with at least 75 m between transects. At the 1m and 4m positions along each transect, a soil corer was used to take five subsamples (depth: ~10 cm) from a 1-m^2 area (Figure 3). The five subsamples were combined into a composite sample and labeled with a unique code. Samples were transported back to the USGS Columbia Environmental Research Center (CERC) in plastic bags.

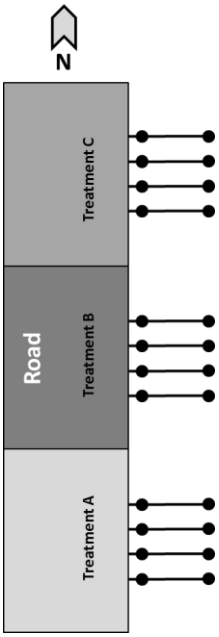

Figure 2. Illustration of soil sample locations at 1m and 4m (circles) along transects (lines) adjacent to road sections treated with dust suppressant products and one untreated control section.

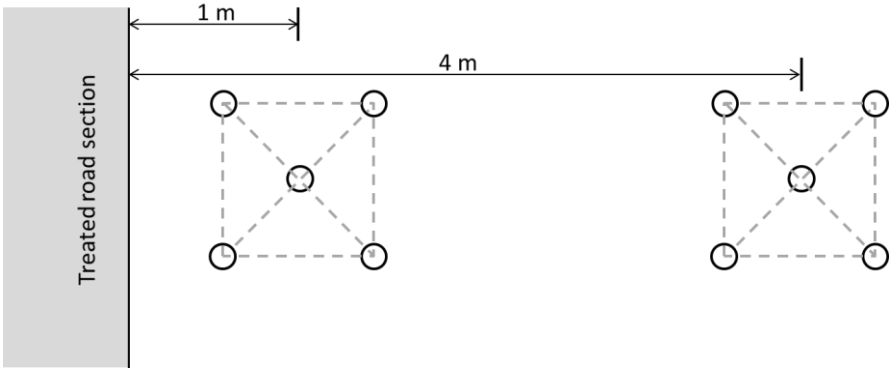

Figure 3. Detail of soil sampling locations at 1m and 4m along one roadside transect. Five subsamples (circles) were taken in a 1-m^2 area and combined into a composite sample for each location.

Analysis

Twenty-six soil samples were transferred to the Environmental Chemistry branch at CERC to be analyzed for the dust suppressant, EnviroKleen®. Prior to analysis, a method development study was conducted for the extraction and isolation of EnviroKleen® from several soil types collected around the CERC campus. The final method involved a 2-step extraction of the soil samples using hexane followed by dichlormethane, with each fraction analyzed separately. The dichloromethane extract required additional cleanup using silica gel chromatography to remove interfering pigments and other materials from the sample.

Analyses were performed using gas chromatography/mass spectrometry (GC/MS) using specific ions characteristic of the EnviroKleen®. The results in Table 1 are a combination of both the hexane and dichloromethane fractions. Initial methods development indicated only a hexane fraction might be necessary; however, the environmentally-incorporated EnviroKleen® was more difficult to extract from the soils, requiring the additional dichloromethane step. Quality Control measures in the study included replicates of randomly selected samples, procedural and matrix (clean soil) blanks, and processing recovery samples.

Table 1. EnviroKleen® measured in roadside soil samples. For each road section, a composite sample was taken along four transects perpendicular to the road, at a distance of 1 m and 4 m from the road's edge. See Figure 1 for map showing locations of EnviroKleen® sections (1) and (2).

Road treatment, distance, and sample	Weight Extracted (g)	EnviroKleen® (µg/g)
Untreated control-1 m		
1	32.91	ND
2	10.18	ND
3	10.07	ND
4	10.12	ND
EnviroKleen® (1)-1 m		
1	10.09	326
2	10.04	1535
3	10.03	114
4	10.04	11.5
EnviroKleen® (2)- 1 m		
1	10.05	21.5
2-R1	10.04	96.2
2-R2	10.11	94.7
3	10.10	207
4	10.08	246
Untreated control- 4 m		
1	10.09	ND
2	10.08	ND
3	10.09	ND
4	10.09	ND
EnviroKleen® (1)- 4 m		
1	10.15	28.9
2-R1	10.09	1.76
2-R2	10.09	2.44
3	10.12	22.3
4	10.10	5.22
EnviroKleen® (2)- 4 m		
1	10.06	4.51
2	10.08	5.71
3	10.05	8.14
4	10.19	12.4
QA/QC - Blanks		
PB031616 Set 1 Hex	10.00	ND
MB031616 Set 1 Hex	10.10	ND
PB032216 Set 2 Hex	10.00	ND
MB032216 Set 2 Hex	10.07	ND
QA/QC – Spike Recovery		Percent recovery
Concentration Steps	-----	95.90%
Silica Gel cleanup	-----	111%
MS031616 Set 1 Hex	10.06	83.10%
MS031316 Set 1 DCM	10.06	ND
MS032216 Set 2 Hex	10.07	81.00%
MS032216 Set 2 DCM	10.07	ND

R1/R2 – Replicate 1 and 2

PB – Procedural Blank (all reagents and materials, no soil matrix)

ND – Not Detected

MB – Matrix Blank (EnviroKleen-free CERC soil)

MS – Matrix Spike (blank CERC soil spiked with EnviroKleen® prior to extraction)

Hex – Hexane fraction

DCM – Dichloromethane fraction