#### **RESEARCH ARTICLE**



# Low-thermal remediation of mercury-contaminated soil and cultivation of treated soil

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#### Abstract

In this study, low-thermal technology was used to treat the mercury contaminated farmland soil from a chemical plant in Guizhou Province, China. A series of field planting experiments were also aimed at determining the content of total and methyl-Hg in crop plants after thermal treatment. The results showed that the mercury concentration in soils was reduced about 70% from 255.74 mg/kg to 80.63 mg/kg when treated at 350 °C for 30 min in engineering-scale experiments, and the treated soil retained most of its original soil. Organic-bound and residual mercury in treated soil were reduced by 64.1 and 56.4% by means of a sequential extraction procedure, respectively. The total and methyl-mercury concentrations in crops decreased significantly, and the degree of soil mercury accumulation to crop roots has been reduced significantly. The total Hg concentrations in potato and corn were lower than the mercury tolerance limits for food in China, and the Hg concentration of radish was close to the limit. The technology provides a more sustainable remediation method for treating mercury-contaminated farmland soil in future engineering applications.

Keywords Low-thermal technology · Mercury · Farmland soil · Cultivation

Mercury (Hg) is a persistent and biologically toxic heavy metal, which cycles through the environment in various forms (Adriano 2001). Generally, the average background concentration of mercury in soil ranges from 0.03 to 0.1 mg/kg with an average value of 0.06 mg/kg. Anthropogenic activities, including Hg mining, smelting, production, and application processes, have mobilized increasing quantities of Hg and is currently the source of an intense worldwide public health concern (Horvat et al. 2003; Clarkson and Magos 2006; Díez 2009; Palinka et al. 2010). More than 90% of Hg entered terrestrial ecosystem (Fitzgerald 1995; Wang et al. 2003) in which the soil was the largest Hg recipient. Several authors

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(Higueras et al. 2006; Millán et al. 2011; Schmid et al. 2003; Sierra et al. 2012) measured total Hg concentrations in surface soils ranging from 0.5 to 8889 mg/kg. In China, the mercury concentrations in some soils are up to 232 times the maximum mercury concentration allowed for soil in China (1.5 mg/kg, National Standard GB15618-1995) (Jiang et al. 2006). The bioavailability and toxicity of Hg in soils are strongly dependent on its chemical speciation (Stein et al. 1996). Soluble Hg salts can be directly taken up by plants and typically serve as the substrate for Hg (II) reduction and methylation during various biological and abiotic processes. Methyl-mercury only represents a small fraction of the total Hg in soils, whereas it is a more toxic species and can be bioaccumulated in the food chains (Qian et al. 2009; Akagi and Nishimura 1991).

Many soil remediation techniques, including phytoremediation (Smolinska and Rowe 2015; Wang et al. 2011; Kiyono et al. 2013), stabilization/solidification (Piao and Bishop 2006; Robles et al. 2014), soil washing (Sierra et al. 2011), thermal treatment (Huang et al. 2011), electrokinetics (Shen et al. 2009), and so on, are applied on the Hgcontaminated soils. Stabilization/solidification is the process of converting Hg into chemical forms that are stable and highly insoluble over wide ranges of pH and redox conditions in soil. But this technology changes soil properties dramatically

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and makes the treated soil unsuitable for agricultural reuse. Plants and soil require long-term monitoring for phytoremediation (Rahimi et al. 2013). Soil washing (ex situ technique) needs high construction costs of the cleaning installation and utilization (Wuana and Okieimen 2011). Electro-kinetic remediation is applicable to different metals, but any heterogeneity of the soil body decreases the effectiveness of this method and considerable acidification of the remediated soil is a side effect of this method (Tahmasbian and Nasrazadani 2012). Thermal treatment, a kind of ex situ remedial technology which involves heating to remove Hg from soil through volatilization, has achieved an acceptable decontamination level when mercury-contaminated soils are treated at temperatures above 600 °C (Chang and Yen 2006; Navarro et al. 2009). However, the high heating temperature requires high-energy cost; moreover, it changes the physical and chemical properties of soil, including increase in soil pH, dehydration of silicate clay minerals, and reduction in the content of organic carbon, thus making the soils unfavorable for agricultural reuse (Kunkel et al. 2006).

This work proposed using low-thermal technology to treat mercury-contaminated farmland soils at 350 °C for 30 min in engineering-scale experiments. Besides, the field planting experiment was conducted to investigate the content of total and methyl-Hg in crop plants before and after thermal treatment. The aim was to study the total Hg and methyl-Hg bioaccumulation in crops after thermal treatment, while the results obtained in this study were expected to provide an environmental friendly technique for treating mercury-contaminated soil in future engineering applications.

# Materials and methods

### Selection and low-thermal treatment of the study soil

The Qingzhen organic chemical plant, situated in the province of Guizhou in Southwest of China (Fig. 1), used Hg as a catalyst during the production of acetic acid in the period from 1971 to 1997 lasting for 26 years. More than 100 t of Hg was released into the Baihua Lake and surrounding farmland. As a result, the Hg concentration of the surrounding farmland was 24~347 mg/kg, and the pollution area was 117.4 ha in the region (Qu et al. 2004). The mercury-contaminated soil was selected to implement low thermal desorption of Hg, and the treated soil was selected to implement the field planting experiment. Test sites 1 and 2 were used as the soil before and after thermal treatment, respectively.

As shown in Fig. 2, low thermal treatment was performed in an engineering-scale combustor with a mercury vapor gas treatment system. The combustor was developed on a scale of 150 kg/h. The combustor was 8-m long with an internal diameter of 0.8 m and was heated by natural gas. At the internal of combustor, a rotating mixing shaft was set up, and in each test, the mercury-contaminated soil was heated evenly under the stirring shaft at 350 °C, and the average treatment time of soil samples in the hot zone was about 30 min. During the treatment, mercury vapor gas would flow into a cooler and condense into liquid mercury at 25 °C, and the cooling water would be used as circulating water. The liquid mercury was collected in a tank, the vapor gas would flow through three-stage-activated carbon adsorption tank to remove uncondensed mercury vapor gas, and the mercury concentration in the exhaust gas met China Integrated Emission Standard of Air Pollutants (12  $\mu$ g/m<sup>3</sup>, National Standard GB16297-1997). Condensed water was the soil moisture during the treatment. At the end of the combustor, condensed water and the soil after repair was collected.

## Field planting of treated soils

The soils before and after the thermal treatment were used as a comparison for the field planting experiment. For the treated soil, in order to cut off mercury pollution from the underlying soil, approximately 10-cm layer of clay was covered as the isolation layer at the bottom of the ground. Subsequently, the soil after thermal treatment was backfilled on the surface of isolation layer, and the laying depth of soil was approximately 30 cm. According to local crop varieties and farming seasons, celery cabbage (*Brassica rapa pekinensis*), napa cabbage (*Brassica campestris L.*), radish (*Raphanus sativus L.*), potato (*Solanum tuberosum L.*), corn (*Zea mays L.*), wheat (*Triticum aestivum L.*) and rice (*Oryza glaberrima*) were cultivated in the soils before and after thermal treatment in the period from 2016 to 2017.

## Sample collection

The surface soil samples  $(0 \sim 20 \text{ cm}, n = 12)$  were collected, and all samples were a composite of at least five subsamples to increase the spatial resolution of sampling. During our sampling campaign, all collected soil samples were stored in the sealed polyethylene bags to avoid cross contamination, and then immediately shipped to the laboratory. All soil samples were air-dried at room temperature, then ground with a glass mortar, and passed through a 100-mesh sieve, and finally stored in the polypropylene bags until analysis. During the crop maturation period, the plant samples (n = 12) were collected from a 1-m<sup>2</sup> area at each sampling site. Each plant sample was a composite of at least ten crop plants to increase the spatial resolution of sampling. After being cleaned with tap water, the individual crop plants were separated into root, stem, leaf, and edible parts. All samples were air-dried, ground to a fine powder in a pre-cleaned food blender, and passed through a 60-mesh sieve, and stored in polypropylene bags until analysis.

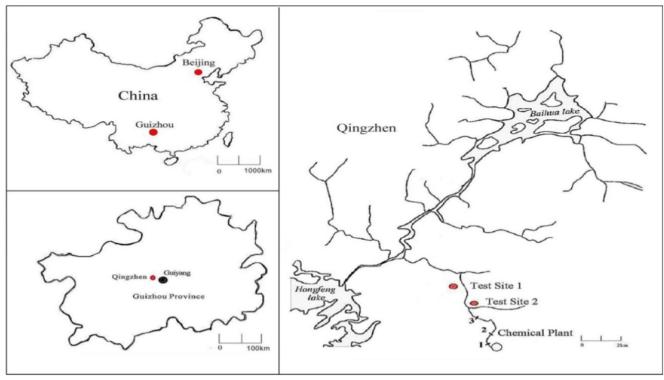


Fig. 1 Location of the experimental area and soil sampling sites

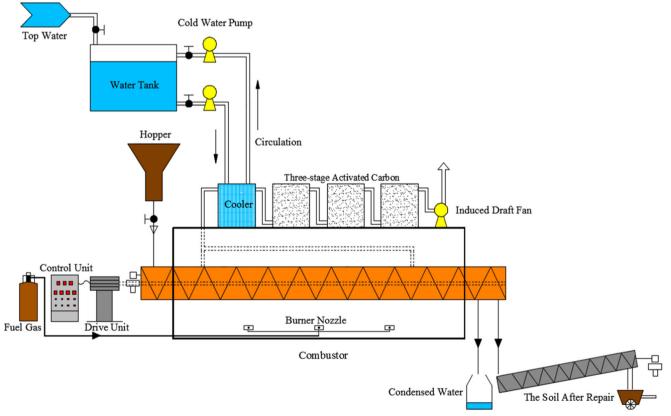


Fig. 2 Simple process flow of low-thermal technology

Indicators	рН	SOM (%)	TN (g/kg)	Alkalytic N (mg/kg)	TP (g/kg)	Available P (mg/kg)	TK (g/kg)	Readily available K (mg/kg)	Soil total porosity %
Before thermal	$7.50\pm0.17$	26.98	3.33±0.66	$100 \pm 5.75$	$0.72 \pm 0.02$	35.1±0.76	$12.3 \pm 0.43$	$1053 \pm 193.5$	76.31
treatment After thermal treatment	$7.61\pm0.02$	26.88	$2.82\pm0.17$	$114\pm5.85$	$0.77\pm0.04$	$85.6 \pm 1.39$	$12.1 \pm 0.71$	471±16.0	63.15

Table 1 Physicochemical properties of soils before and after the thermal treatment

All values are expressed as means  $\pm$  S.D. (n = 12). SOM, soil organic matter; TN, total nitrogen; TP, total phosphorus; TK, total potassium

# Analytical methods

Table 2Residual mercuryspeciation of soil before and after

thermal treatment

#### **Total Hg analysis**

The Hg concentrations were measured with an atomic fluorescence spectrometer (AFS) equipped with a flow hydrogen generation system (AFS-640, Beijing Ruili Instrument Co., China). Quality control was exercised by using the method blanks and certified reference materials. The standard soil or vegetable samples were used to accomplish the quality assurance/quality control (QA/QC). The average total Hg concentration of the geological standard of GBW07429 was  $0.096 \pm 0.001$  mg/kg, which was comparable with the certified value of  $0.094 \pm 0.004$  mg/kg. The average total Hg concentration of the geological standard of GBW100014 was  $0.010 \pm 0.001$  mg/kg, which was comparable with the certified value of  $0.011 \pm 0.002$  mg/kg. Meanwhile, the recoveries on matrix spikes of total Hg in the soil and plant samples were in the range of  $91 \sim 107\%$  and  $95 \sim 106\%$ , respectively.

## Methyl-mercury analysis

Methyl-mercury was measured using a combination technology of purging and trapping gas chromatography-cold atomic fluorescence (PT-GC-CAF). Quality control was exercised by using the method blanks and certified reference materials. A standard sediment sample ERM-CC580 and fish sample ERM-CE464 were used to accomplish QA/QC, and the average methyl-mercury concentrations of the geological standards of ERM-CC580 and ERM-CE464 were 71 ± 4 µg/kg and 5.5 ± 0.17 mg/kg, which were comparable with the certified values of 70.68 ± 6.25 µg/kg and 5.13 ± 0.34 mg/kg, respectively. The repeatability of the same digested sample for methyl-mercury determination was over 10%. Meanwhile, the recoveries on

Soil samples	Item	Total Hg	F1	F2	F3	F4	F5
Before thermal	Means (mg/kg)	255.74	0.133	0.087	0.160	140.22	58.84
treatment	Standard deviation (mg/kg)	7.17	0.018	0.006	0.015	5.19	2.70
	Minimum value (mg/kg)	243.29	0.115	0.076	0.141	134.06	55.39
	Maximum value(mg/kg)	263.11	0.163	0.094	0.177	146.29	62.23
	Coefficient of Variation (%)	2.80	13.53	6.90	9.38	3.70	4.59
After thermal	Means (mg/kg)	80.63	0.204	0.052	0.131	37.62	24.83
treatment	Standard deviation (mg/kg)	2.91	0.019	0.009	0.011	2.93	1.59
	Minimum value (mg/kg)	76.55	0.187	0.039	0.117	32.67	22.12
	Maximum value(mg/kg)	85.43	0.234	0.062	0.143	40.64	26.45
	Coefficient of Variation (%)	3.61	9.31	17.31	8.40	7.79	6.20
Removal rate (%)		68.47	- 53.38	40.23	18.13	73.17	57.80

F1, water-soluble fraction; F2, special absorbed fraction; F3, oxide-bound fraction; F4, organic-bound fraction; F5, residual fraction. n = 12

Crops	Total Hg (mg/kg)		Methyl-Hg (µg/kg)	Tolerance	
	Before thermal treatment	After thermal treatment	Before thermal treatment	After thermal treatment	limit of total Hg (China)
Celery cabbage Napa cabbage	$\begin{array}{c} 0.0621 \pm 0.0106 \\ 0.0780 \pm 0.0154 \end{array}$	$\begin{array}{c} 0.0585 \pm 0.0137 \\ 0.0707 \pm 0.0111 \end{array}$	$\begin{array}{c} 1.04 \pm 0.054 \\ 1.121 \pm 0.101 \end{array}$	$\begin{array}{c} 0.825 \pm 0.051 \\ 0.905 \pm 0.023 \end{array}$	0.01
Radish	$0.1072 \pm 0.0213$	$0.0171 \pm 0.0042^{**}$	$0.916 \pm 0.021$	$0.113 \pm 0.012^{**}$	
Potato	$0.0086 \pm 0.0023$	$0.0042 \pm 0.0012 *$	$0.252 \pm 0.053$	$0.245 \pm 0.049$	
wheat Rice Corn	$\begin{array}{c} 0.0543 \pm 0.0122 \\ 0.0660 \pm 0.0135 \\ 0.0080 \pm 0.0022 \end{array}$	$\begin{array}{c} 0.0310 \pm 0.0069 * \\ 0.0305 \pm 0.0075 * \\ 0.0045 \pm 0.0016 * \end{array}$	$\begin{array}{c} 1.961 \pm 0.193 \\ 14.52 \pm 1.39 \\ 1.939 \pm 0.478 \end{array}$	$\begin{array}{c} 1.249 \pm 0.087 * \\ 6.48 \pm 1.89 * * \\ 0.925 \pm 0.216 * \end{array}$	0.02

Date in table are means  $\pm$  S.D. (n = 12). \*Significant correlation at the level of P < 0.05, \*\*Significant correlation at the level of P < 0.01

matrix spikes of methyl-mercury in the soil and plant samples were in the range of 94~110% and 93~107%, respectively.

#### Hg speciation analysis

To better understand the effect of low-thermal treatment on the mercury-contaminated soil, sequential extractions of soils before and after thermal treatment were conducted. Mercury in soil samples before and after thermal treatment were separated into soluble and exchangeable fraction (F1), special absorbed fraction (F2), oxide-bound fraction (F3), organic-bound fraction (F4), and residual fraction (F5) (Biester and Scholz 1997). The concentrations of all Hg species were measured with an atomic fluorescence spectrometer (AFS).

#### **Data analysis**

The software package used to perform the statistical analysis was SPSS 12.0 for Windows. One-Way ANOVA analysis of total and methyl-Hg concentrations in soil, edible parts of crops and other organs for the before and after thermal treatments was also performed. Data were presented as mean and standard deviation, and statistical significance was considered at the 0.05 level.

# **Results and discussion**

#### Changes in soil physicochemical properties

In this study, the Hg concentration of the soil was  $255.74 \pm 7.17 \text{ mg/kg}$  before the thermal treatment and  $80.63 \pm 2.91 \text{ mg/kg}$  after the thermal treatment. The physicochemical properties of soils before and after the thermal treatments are listed in Table 1. Soil pH values remained near neutral (pH 7.6) and SOM (soil organic matter) remained near 26.88% when treated at 350 °C for 30 min. There was

no significant difference in pH and SOM between treated soil and background soil. The main reason is that the high concentration of refractory organic matter in study soil was attributed to the organic chemical plants. Previous research has shown that the pH value of treated soil was always kept lower than the pH value of the background soil regarding soil pH variation with temperature (Sierra et al. 2016). The change in pH was attributed to the displacement of H<sup>+</sup> from exchange sites of clay and OM by base cations released by heating effect. SOM is mineralized releasing CO<sub>2</sub> that is easily transformed in HCO<sub>3</sub><sup>-</sup> (Terefe et al. 2008).

Regarding the available P and the readily available K differences between treated soil and background soil, the available P increased approximately 1.5 times, and the readily available K was reduced by 60.4%. During the thermal treatment process, the available P and the readily available K migrated and transformed with changes in temperature and moisture (Zhang et al. 2012). When temperature was increased, the fixed P was released and transformed into the soluble P, the readily available K was transformed into the fixed K. However, the other physicochemical indicators were not much affected. Overall, the result indicated that the 350 °C-treated soil retained most of its original soil physiochemical properties, suggesting the treated soil may be suitable for reuse on agricultural land.

#### Changes in soil mercury speciation

As shown in Table 2, the summed values of Hg concentrations in all Hg species were generally lower than the total Hg concentrations directly measured for all soil samples, and the extraction rate was approximately 78.0%. 40.33% of F2, 18.13% of F3, 73.17% of F4, and 57.80% of F5 were removed when soil sample was treated at 350 °C for 30 min. The organic-bound and residual fractions were still the two main chemical species of Hg in soil after thermal treatment. However, the soluble and exchangeable fraction increased

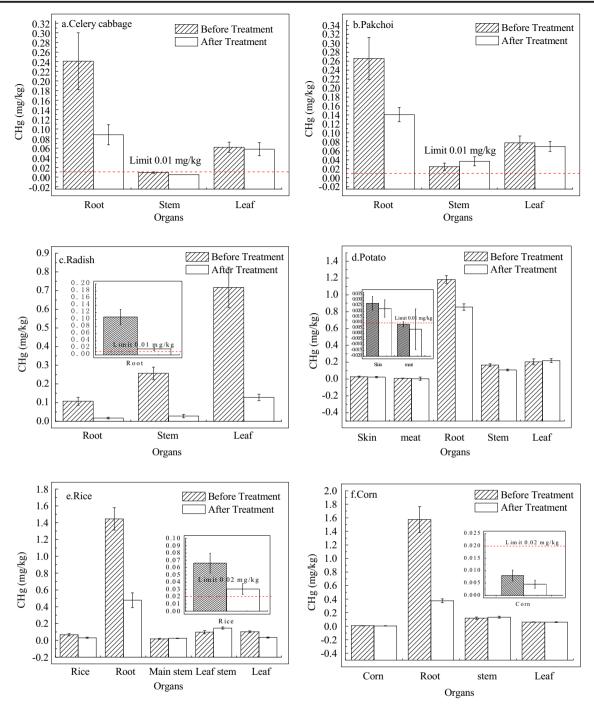


Fig. 3 Total mercury distribution in different organs of crops

by 53.38% observably in soil after the thermal treatment due to the gaseous Hg settled back into the soil with a change in temperature during the low-thermal treatment process. Previous research has shown a significant increase of soluble and exchangeable Hg concentration at 220 and 280 °C (Sierra et al. 2016). Some authors measured that the cultivation of the study soils heated to those temperatures would be a risk due to the fact that crops could uptake this Hg (Millán et al. 2013; Sierra et al. 2012). Thus, the field planting experiment was conducted to investigate the exposure risk of total Hg and methyl-Hg in treated soil and the effect on crops.

# Bioaccumulation of total and methyl-Hg in crops

The primary risk of Hg exposure to terrestrial vertebrates, including humans, is through the ingestion of contaminated food and agricultural products (Doty 2008). The statistical summaries of the total and methyl-Hg concentrations in the

edible parts of crops are listed in Table 3. Results showed that the Hg concentration in the edible part of radish was significantly reduced by 84.05% (p < 0.01) after thermal treatment. Moreover, the Hg concentrations in the edible parts of potato, wheat, rice, and corn decreased significantly (p < 0.05). At the same time, no significant (p > 0.05) differences were observed between Hg concentrations in the edible parts (leaves) of celery cabbage and napa cabbage before and after thermal treatment. Recent studies have shown that the ability of different crops to absorb Hg in soil follows the order: leaf vegetables > root vegetables > fruit vegetables (Huang et al. 2011; Lindberg et al. 1979). This suggested that the mercury in leaf vegetables was still mainly from the soil because the Hg content of treated soil was still up to 80.63 mg/kg.

In this study, the Hg concentrations of potato and corn were lower than the Hg tolerance limit for food in China. After thermal treatment, the Hg concentration of radish was close to the limit (GB 2762-2012). Potatoes are rich in selenium, and studies have shown that selenium concentrations in the potato plants show unimodal curve change in the whole cultivation process. The selenium content of leaf, stem, and tuber organs in the whole growth period averaged 0.137, 0.063, and 0.031 mg/kg, respectively, and the selenium concentrations ranged between 0.025 and 0.042 mg/kg in tuber during the harvest period (Xing et al. 2012). At the same time, selenium has an inhibitory effect on Hg toxicity in plants (Wang et al. 2002; El-begearmi et al. 1977).

The mean methyl-Hg concentration of the soil was  $174.2 \pm 6.14 \ \mu g/kg$  and  $21.9 \pm 1.01 \ \mu g/kg$  before and after thermal treatments, respectively, showing that the removal rate of methyl-Hg in soil reached 87% after thermal treatment. In this case, the methyl-Hg concentration was significantly reduced by 87.66% in radish and 55.3% in rice (p < 0.01). Moreover, the methyl-Hg concentrations in wheat and corn decreased significantly (p < 0.05). On the other hand, the means were not significant (p > 0.05) in other edible parts of crops between before and after thermal treatments.

#### Total Hg distribution in crop plants

The statistical summaries of total mercury concentrations in different organs of crops are shown in Fig. 3. The root of radish was regarded as main edible part, in which the mercury content was about 5~40% of other crop roots. Mercury distribution in the radish plant was in the following order: leaf> stem > root, and mercury content of leaf and stem were significantly decreased after the remediation treatment. Besides, mercury distributions in other crops were mainly in the following order: root > leaf> stem, and the roots were suffered serious persecution from soil Hg. However, after the thermal treatment, mercury contents of all roots were significantly decreased two~five times and other organs were not significantly different. Total Hg concentrations of plant roots were

higher than total Hg concentrations of ground parts of the plant (Godbold 1991). Many studies showed that the root Hg concentrations of plants were related to the soil Hg concentrations (Frescholtz et al. 2003; Ericksen et al. 2003; Millhollen et al. 2006). Overall, the degree of soil mercury accumulation to crops roots had been significantly reduced after the thermal treatment.

# Conclusion

In this study, approximately 70% Hg was removed from the contaminated soil by the low-thermal technology, and the treated soil retained most of its original soil physiochemical properties. The organic-bound and residual fractions of Hg were reduced by 73.17 and 57.80%, respectively. Especially, the removal rate of methyl-Hg in soil reached 87%. However, the soluble and exchangeable fraction increased by 53.38% observably in soil after the thermal treatment. The field experiment shows that the total and methyl-Hg concentrations of crops decreased significantly, and the degree of soil mercury accumulation to crops roots have been reduced significantly. The Hg concentrations of potato and corn were lower than the Hg tolerance limits for food in China, and the Hg concentration of radish was close to the limit (GB 2762-2012), thus the treated soil was suggested to cultivate tuber, high strain and root vegetable crops.

In conclusion, the low-thermal technology provides a more sustainable remediation method for treating mercury contaminated farmland soil in future engineering applications, but it is very essential to have a long-term monitoring on the cultivation of treated soil.

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