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ORIGINAL ARTICLE

Preliminary assessment of cement kiln dust in solidification and stabilization of mercury containing waste from a chlor-alkali unit

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Abstract A low-level mercury containing hazardous waste with a mercury concentration of 22.0 ± 5.00 ppm generated at a chlor-alkali unit was solidified and stabilized using cement kiln dust (CKD) and other binding agents. Similar in many aspects to cement, CKD is a waste byproduct of cement manufacturing process and is considered a waste material although in some cases it is partially reused. Application of CKD as a binding agent in solidifying and stabilizing mercury in the waste was preliminarily examined. The results indicated that addition of about 36.0 % by dry mass of CKD to the waste led to reduction of mercury concentrations to less than the strict land disposal restriction (LDR) of 25.0 ppb in toxicity characteristic leaching procedure extracts set by the US Environmental Protection Agency. Using CKD for solidifying/stabilizing mercury can be advantageous as it can be partially consumed in this process rather than being discarded as a waste material. However, the volume of the resulting solidified/stabilized waste may not be favorable for land disposal or onsite storage. Addition of less than 1.00 % of other chemicals such as lime, soda ash, sulfur and sodium metasilicate individually to the mixture resulted in about 12.0 % reduction in CKD requirement.

Keywords Mercury · TCLP · Chlor-alkali · Cement kiln dust · Solidification · Stabilization

Introduction

Elemental mercury (Hg^0) is unique among metals because it is a liquid at room temperature with a low boiling point, and high vapor pressure that can react to form compounds in either the (Hg^{1+}) or (Hg^{2+}) oxidation states [1]. Mercury originating from human activities can be transported in the environment, depending on the form emitted and the receiving environment, before depositing in water. In aquatic ecosystems, mercury can be environmentally transformed into the organic form of mercury, methyl mercury, which can bioaccumulate and biomagnify through food webs, and is highly toxic [2]. Mercury can pose serious health issues and its emission to environment needs to be controlled [3, 4]. Various industrial wastes may contain different concentrations of mercury and its compounds restricting land disposal [5]. Among industries with mercury emission is the chlor-alkali process producing chlorine and caustic soda. The mercury-cell chlor-alkali process is an electrochemical process used for generating chlorine gas and sodium hydroxide [6]. In this process, elemental mercury is used as the cathode material, and chlorine is generated from the brine (i.e., sodium chloride). During this process, elemental sodium is produced at the cathode and forms an amalgam with the elemental mercury. The mercury–sodium amalgam is decomposed in water, forming sodium hydroxide and the elemental mercury which is recycled back into the process. During the manufacturing process, some elemental mercury is oxidized and forms mercury chloride compounds and mercury sulfides. Caustic wastewater sludge from this process typically contains mercury(I) and mercury(II) chlorides, and elemental mercury [7].

Disposal of mercury containing waste is restricted based on leachable concentrations estimated through toxicity

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characteristic leaching procedure (TCLP) test. The waste is considered hazardous if the TCLP concentration of mercury exceeds 0.20 mg/L. A more restrictive measure is set by the US EPA for a low-level mercury containing waste namely land disposal restriction (LDR) requirement which is set to be 25.0 µg/L [8, 9]. Mercury concentrations exceeding these levels should be treated either through removing mercury and its compounds from the waste or immobilizing them within the waste structure [8]. The former can be achieved through a number of chemical and thermal methods and the latter through solidification and stabilization processes (S/S). Solidification/stabilization generally comprises both chemical binding and physical encapsulation of waste to a solid product aiming at achieving low leachability of contaminants including mercury [8]. A number of materials have been used to solidify and stabilize mercury in different wastes with the main component being various types of cement [5, 10–15]. Portland cement as a binding agent has been more frequently used to treat contaminated material than any other S/S binding agent because of its binding ability resulting in reduced permeability [8, 16, 17]. Other materials including but not limited to limestone, sodium hydroxide and sulfur have also been used in combination with cement to either immobilize the contaminant within the waste or to enhance chemical or physical binding of the contaminant [10].

Solidification/stabilization processes have proven to be successful in immobilizing a number of heavy metals including but not limited to Pb, Cd and Cr. However, it is more difficult to reduce the leachability of mercury using conventional S/S processes [18]. This necessitates the use of additional binding agents such as sulfide and phosphate, sulfur polymer cement, polyester resins or polysiloxane compounds [8, 14, 15, 19, 20]. Mercury sulfide (i.e., HgS) has the lowest solubility in water of about 10 µg/L while $HgCl_2$ has the highest solubility of 70.0 g/L [1]).

Using other waste materials as the binding agent in S/S of mercury containing waste can result in reducing downstream waste management efforts and the associated costs. Cement Kiln Dust (CKD) that is a waste byproduct of cement production has also been used in a limited number of cases to stabilize various metals [21]. CKD is a fine powder like material similar to cement that includes the dust generated in clinker, raw material, alkaline sulfate rich ash, halides and some volatile materials [21, 22]. CKD with a specific gravity of 2.6–2.8 has a relatively large specific surface of 4600–14,000 cm^2/g and a maximum particle size of 0.30 mm [22, 23]). Chemically CKD is mainly composed of CaO, Fe_2O_3, SiO_2 and Al_2O_3 [22, 23]. When mixed with water, pH values of above 12 have been reported [22]. Very low level of trace elements has been found in CKD [24]. As a waste material, CKD can be recycled in various ways; however, the main recycling method is to return it to the kiln, although not quite viable in some cement kilns [21, 22].

Using CKD in S/S processes to minimize heavy metal leachability has not been widely experienced [10]. Having similar properties to cement, CKD was assumed to be a suitable binder to solidify and stabilize mercury containing waste generated at a local chlor-alkali unit in south of Iran. No similar experience was found in the literature to the extent reviewed. Therefore the main objective of this research was to perform a preliminary examination of the capability of CKD in solidifying and stabilizing mercury within industrial waste. Furthermore, enhancement of S/S performance of CKD using typical binding agents and its effect on reduction of the product volume were also examined preliminarily. CKD sampled from a local cement manufacturer was used solely and in combination with sulfur, sodium metasilicate, limestone and sodium hydroxide to solidify and stabilize a low-level mercury containing waste. The mercury containing waste used in this study is currently being disposed of in a landfill equipped with a high-density polyethylene geomembrane laid over a compacted clay liner. Possible leaching of mercury to the local groundwater system was the main concern.

Materials and methods

Mercury containing waste

The waste in this study was a filter pressed brine mud with about 25.0 % by weight of NaCl and a moisture content of about 47.0 % by weight generated at a local chlor-alkali unit. The waste was sampled and was transferred to the lab in mixed form at 4 °C with no preservatives added. The waste sample was kept at 4 °C throughout the experiment and was brought to room temperature prior to performing the tests. The general composition of the waste is summarized in Table 1. Concentration of mercury within the waste was measured in triplicate according to US EPA methods 7470A, using a UNICAM 919 atomic absorption spectrophotometer. Concentrations of different species of mercury were not measured since the total concentration of mercury was set as a target for the level of stabilization.

S/S agents

The main S/S agent used in this study was CKD obtained from a local cement manufacturer in the form of a composite sample. In order to examine the effect of other binding agents enhancing the stabilization of mercury in the waste, sulfur, NaOH, $CaCO_3$ and sodium metasiliacate (Na_2SiO_3) were added along with CKD. Cubic samples of

Table 1 General composition of the filter pressed brine mud containing mercury

Brine mud	% by dry mass
NaCl	13.9
Water	36.0
$BaSO_4$	31.5
$CaCO_3$	10.0
$Mg(OH)_2$	5.60
NaOH	3.00
Na_2CO_3	
Na_2SO_4	
Flocculant	
Total mercury (mg/kg)	22.0 ± 5.00

50 mm × 50 mm were prepared in accordance with Refs. [25, 26] containing various percentages of the selected binding agents. The composition of the S/S samples in terms of binding agent content is summarized in Tables 2 and 3. Distilled and deionized water was used in all mixtures. Due to the paste like form of the waste, the specimens were made up in a few steps by taking portions of the waste and CKD and spreading on a flat surface. Water was added gradually to the mix and the content were mixed using spatula. The mix was filled in a prefabricated cubic container made of plexiglass and was gently compacted in layers after being de-aired using a vibrator.

One of the issues observed when cubic S/S samples were made was formation and accumulation of different amounts of crystalline salt on top of the samples which could be associated with the relatively high salt content of the waste. Qualitatively it could be concluded that the higher the percentage of the waste in the sample, the greater the salt accumulation on top of the sample. Formation of salt layer, although not anticipated at the planning stage of the experiments, was found to be important in terms of mercury content which could have a high leachability due to very high solubility of the salt presumably containing $HgCl_2$ amongst others. The salt was tested for mercury content and leachability. Figure 1 shows some of the samples with and without the salt layer formed.

Toxicity assessment using leachability test

Toxicity tests in general can lead to the determination of either potential hazard of the toxic contaminant or its ecotoxicological properties. The former is accomplished by leaching the contaminant using water and other mildly acidic solutions representing atmospheric precipitation. Leachability tests are aimed at evaluating the potential

Table 2 Composition of S/S samples using different percentages of CKD to stabilize mercury in the waste

Sample ID	Waste		CKD		Water	
	% by weight	Mass (g)	% by weight	Mass (g)	% by weight	Volume (ml)
S1	66.7	180	7.40	20.0	25.9	70.0
S2	59.3	160	14.8	40.0	25.9	70.0
S3	50.9	140	21.8	60.0	27.3	75.0
S4	36.4	100	36.4	100	27.3	75.0
S5	30.5	80.0	45.8	120	23.7	62.0
S6	22.9	60.0	53.4	140	23.7	62.0
S7	15.3	40.0	61.1	160	23.7	62.0
S8	7.10	20.0	64.3	180	28.6	80.0
S9	3.50	10.0	66.7	190	29.8	85.0
S10	0.00	0.00	70.2	200	29.8	85.0

Table 3 Composition of S/S samples using different percentages of CKD and other stabilizing agents to stabilize mercury in the waste

Sample ID	Waste		CKD		Water		Sulfur		NaOH (30 %)		$CaCO_3$		Na_2SiO_3	
	% by mass	Mass (g)	% by mass	Mass (g)	% by mass	Volume (ml)	% by mass	Mass (g)	% by mass	Volume (ml)	% by mass	Mass (g)	% by mass	Mass (g)
S11	48.7	134	24.0	66.0	27.3	75.0	0.61	1.30	1.43	4.00	–	–	–	–
S12	62.2	168	11.9	32.0	25.9	70.0	0.70	1.70	1.81	5.00	–	–	–	–
S13	48.7	134	24.0	66.0	27.3	75.0	0.46	1.30	–	–	1.43	4.00	–	–
S14	62.2	168	11.9	32.0	25.9	70.0	0.61	1.70	–	–	1.81	5.00	–	–
S15	36.4	100	36.4	100	27.3	75.0	–	–	–	–	–	–	6.14	18.0

(S10) (S5) (S14)

Fig. 1 Some S/S samples with and without formation of salt layer

hazard of the contaminant without considering their exposure pathways. Where leachability tests are employed, the associated human health risk can be evaluated through a site-specific modeling of exposure pathways. In cases where the waste is to be disposed of in a landfill, modeling focuses on assessment of potential or actual risks associated with contaminated groundwater uptake by humans. An example of such models is Industrial Waste Management Evaluation Model (IWEM) [27].

US EPA has set a leaching test for the assessment of hazardous wastes namely Toxicity Characteristic Leaching Procedure (TCLP) for assessment of hazardous wastes [28].

Since the mercury containing waste in this study was to be landfilled, current LDR requirement set by the US EPA [28] was considered as the leachability criterion and therefore TCLP test was employed to determine the potential mobility of mercury in a waste under simulated landfill condition as a standard US EPA regulatory test (40 CFR 261.24, SW-846 Method 1311). For the TCLP tests, the S/S samples were size reduced to pass a 9.5 mm sieve. Then, the samples were exposed to a 20-fold larger volume of acetate buffer. After being tumbled for 18 h, the mixture was filtered through a 0.8 μm filter. The mercury limit to pass the TCLP test is 0.20 mg/L. A TCLP tumbler device was manufactured in accordance with Method 1311 capable of accommodating six polyethylene bottles of 1 l volume and tumbling for 18 h. TCLP tests were repeated three times for each sample. A blank test was also run in order to account for any mercury concentration resulting from the bottles with no waste in the extracting solution. Mercury was not detected in the blank solutions.

First mercury content of the waste was measured in accordance with US EPA SW-846-method 7471A and then the waste was subjected to TCLP test with no prior extraction since the solid content of the waste was greater than 0.5 %. The salt layer formed on top of some of the samples was also tested for mercury content. The concentration of mercury in the salt formed on the samples solidified and stabilized using CKD was 3.00 ± 3.06 μg/L. Since this concentration was well below the LDR levels, TCLP test was not performed for the salt layer.

Table 4 Average concentration of mercury in TCLP tests of S/S samples with different binding agents

Sample ID	TCLP concentration of mercury (μg/L)	Standard deviation
S1	47.0	2.10
S2	43.0	5.00
S3	39.0	2.10
S4	23.0	3.50
S5	14.0	2.10
S6	12.0	2.10
S7	8.00	1.00
S8	6.00	1.50
S9	2.00	1.50
S10	<1.0	–
S11	11.0	2.60
S12	18.0	3.00
S13	9.00	2.00
S14	20.0	2.50
S15	4.00	3.50

Results and discussion

TCLP concentration of mercury for untreated waste was 301 ± 15.1 μg/L which exceeds the LDR requirement by over one order of magnitude, necessitating treatment of the waste. The results of TCLP tests for solidified and stabilized waste using various proportions of S/S agents are presented in Table 4. Using CKD as the sole biding agent, the TCLP concentrations of mercury were well below the 200 μg/L level as set by the US EPA [8, 9]. However, to meet the LDR of 25.0 μg/L concentration of mercury, more than 35 % of CKD was required.

As shown in Fig. 2, TCLP concentration of mercury reduced with increasing percentage of CKD added to the waste. The relationship between the percent CKD added to the waste and the TCLP concentration of mercury was linear with a R^2 value of 0.98. If CKD is used as the only binding agent, the waste examined in this study can meet the LDR in terms of mercury concentration by adding 36.7 % by dry mass of CKD. However, this proportion of

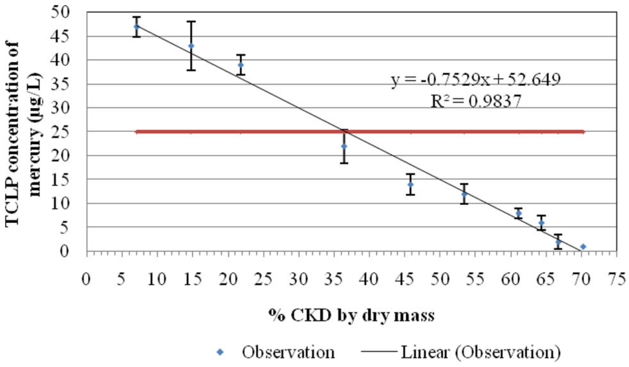

Fig. 2 TCLP concentration of mercury at different percentage of CKD added to the waste

CKD can result in relatively higher final volume of solidified and stabilized product. This can be considered a drawback if the waste is to be landfilled or stored. Thus other binding agents were also examined in terms of their contribution to overall volume reduction.

As shown in Fig. 3, addition of small amounts of other binding agents including sulfur with limestone and sodium hydroxide as well as sodium metasilicate resulted in much lower mercury concentrations in TCLP extracts at lower CKD proportions. As an instance adding about 6.14 % sodium metasilicate alone to the sample containing 36.0 % CKD (S15) resulted in a reduction in mercury concentration to about 4.00 µg/L which is less than 20.0 % of mercury concentration when only 36.0 % CKD (S4) is added to the waste. Samples with about 24.0 % CKD showed more promising concentrations of mercury in TCLP extracts when less than 1 % sulfur was added along with less than 2 % limestone or sodium hydroxide (S11 and S13). The resulting mercury concentrations of 9.00 and 11.0 µg/L were considerably lower compared to the extracts from the samples with only 36.0 % CKD. In terms of efficiency of mercury immobilization, comparing S4 and S15 both containing about 36.0 % CKD, adding about 6.14 % Na_2SiO_3 as a single additive resulted in about 82.0 % reduction of TCLP concentration. Comparing S3 to S11 and S13 having CKD content of 22.0–24.0 %, indicated that adding sulfur and limestone reduced the TCLP concentration by about 77.0 % whereas adding sulfur and sodium hydroxide resulted in about 72.0 % reduction in TCLP concentration. Adding a combination of sulfur and sodium hydroxide as well as sulfur and limestone resulted in a lower efficiency of about 54.0–58.0 % in terms of reducing TCLP concentration when S2 is compared to S12 and S14. This preliminary comparison indicates that sodium metasilicate alone can be quite effective in reducing the mobility of mercury. However, adding sulfur along with either limestone or sodium hydroxide can result in significant reduction in mobility of mercury, yet with lower efficiency compared to adding sodium metasilicate alone. The number of tests as discussed above might not be adequate for a general conclusion on the effect of additives on immobilization of mercury within the solidified/

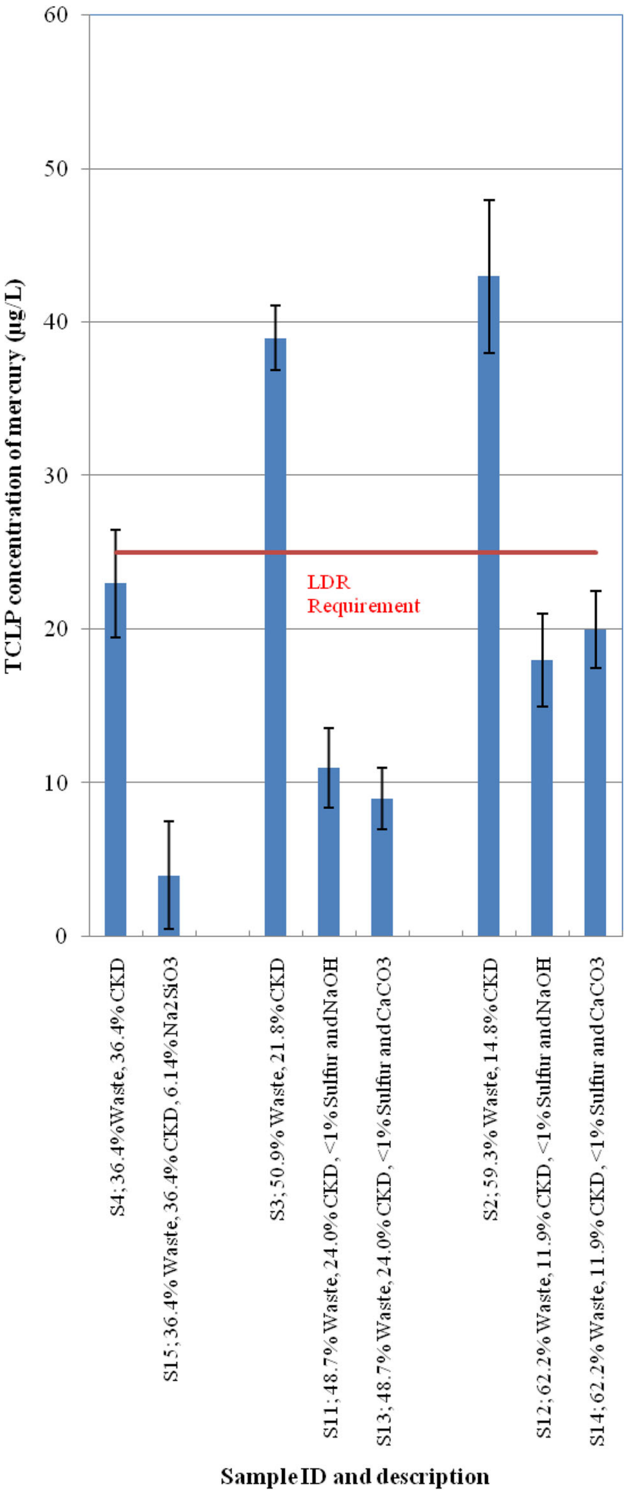

Fig. 3 TCLP concentration of mercury at different percentages of CKD along with other binding agents

stabilized waste, however, it can be concluded that each of the additives and in particular sodium metasilicate and sulfur were considerably effective in reducing the TCLP concentration of mercury.

The key role of additives including sulfur, sodium metasilicate, limestone and sodium hydroxide in reducing the leaching of mercury lies in the relatively low solubility of chemical products. Sulfur when reacting with Hg^{2+} in the presence of a base such as sodium hydroxide or limestone can result in very insoluble mercury sulfide through Eq. 1 [29].

$$Hg^{2+}_{(aq)} + HS^{-} \rightarrow HgS_{(s)} + H^{+} \quad (1)$$

Sulfur is added to the waste at 0.5–3 % by weight, preferably 1 % by weight of the waste. This needs to be done in the presence of a strong base like sodium hydroxide at small portions of about 0.3–10 % by weight of the waste. In this study, sulfur concentrations of less than 1 % along with over 1 % sodium hydroxide were found to be adequate for mercury precipitation. However, excess alkali or alkaline sulfide will increase the solubility of the mercuric sulfide; thus only a small excess of soluble sulfide salt is used for precipitation [30]. The formation of highly insoluble $HgS_{(s)}$ with a solubility of about 12.5 µg/L, either as red cinnabar or the black mineral metacinnabar, is the often cited process for removal of Hg^{2+} from the water column and sediments [31].

An alternative method for precipitation of heavy metals is to form metal silicate through reaction with sodium metasilicate (Na_2SiO_3). Mercury reacts with sodium metasilicate to form mercury silicate with a very low solubility through the following reaction.

$$Hg^{2+} + Na_2SiO_{3(aq)} \rightarrow HgSiO_{3(s)} + 2Na^{+}_{(aq)} \quad (2)$$

The binding agents resulting in partial immobilization of mercury in the solidified/stabilized specimens in general react with mercury resulting in precipitates of different solubility within the solidified/stabilized specimens. While solubility of mercury chloride is about 7×10^{7} µg/L, solubility of mercury sulfide is reportedly 10–12.5 µg/L and mercury silicate is considered insoluble in water [1, 31].

Conclusion

A mercury containing waste was solidified and stabilized using CKD as the main binding agent with and without small amounts of other binding agents including lime and soda ash with sulfur and sodium metasilicate. The results indicated that the addition of about 36.0 % by dry mass of CKD as the sole solidifying/stabilizing agent to the waste led to reduction of mercury concentration to less than the strict LDR requirement of 25.0 µg/L in TCLP extracts. Using waste materials such as CKD in solidifying and stabilizing mercury is advantageous as some of the waste CKD is used in the stabilizing process rather than being disposed of in land. However, the volume of the resulting solidified and stabilized waste may not be favorable for land disposal or onsite storage, when CKD is used with no other chemical binding agent. Addition of less than 1 % of other chemicals such as limestone, sodium hydroxide, sulfur and sodium metasilicate to the mixture indicated that smaller volumes of stabilized waste can be achieved through reduction in CKD requirement. It was shown that addition of sulfur and sodium hydroxide (S11) and sulfur and limestone reduced the amount of CKD to about 24.0 % by mass resulting in mercury concentration in the TCLP extracts to about 11.0 and 9.00 µg/L, respectively. Furthermore, adding sulfur and sodium hydroxide or sulfur and limestone in samples with only 11.9 % by mass of CKD (S12 and S14, respectively) resulted in mercury concentration of about 18.0 and 20.0 µg/L, respectively.

Using CKD in solidification and stabilization of mercury in hazardous waste was not cited in the literature to the extent reviewed. The key question on the capability of CKD to solidify and stabilize mercury was answered in this study. However, it should be noted that using CKD can have two major drawbacks. One of the drawbacks can be increased volume of the solidified product. This can be resolved by addition of other chemical binding agents such as sulfur as tested in this study as described above. Another drawback of using CKD could be unknown longevity of the product. Further research is required to assess the longevity of the product through accelerated aging experiments. Depending on the conditions the solidified/stabilized product would be subjected to, accelerated ageing of the specimens is required at elevated temperatures. The final product can typically be landfilled or used as construction material. In either case, the product might be exposed to immersion in water or leachate, air and sunlight. These conditions need to be simulated through bench or pilot scale accelerated ageing tests. This can be considered a vital part of future research. In addition, accumulation of salt on top of some samples may have an effect on physical properties of the specimens influencing their long term performance. Salt deposition can increase the surface area exposed to environmental conditions and therefore increase the chance of leachability. It can also have an effect on the mechanical strength of the specimen. This also requires further research.

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