Assessment of the feasibility of anaerobic composting for treatment of perchlorate – contaminated soils in a war zone

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ABSTRACT

Aims: The objectives of this study were to determine the perchlorate concentrations in surface soils and assess feasibility of anaerobic bioremediation in full-scale for perchlorate-contaminated soils in a war zone.

Materials and Methods: Fifteen samples of surface soil were collected using a composite sampling method in the study area. The soil samples, after extraction and preparation, were analyzed by ion chromatography. Anaerobic composting technique (soil excavation, mixing with manure, transfer into treatment cell and cover with a 6-mil high-density polyethylene liner) considered to cleanup perchlorate-contaminated soil in a war zone.

Results: The concentration of perchlorate in the soil surface samples ranged from 3 to 107.9 mg/kg, which is more than State advisory levels for residential and protection of domestic groundwater use pathway. This study indicates that technologies, skills, experience, raw materials (manure), lands, and machinery needed for implementation of full-scale composting, are available in the study area.

Conclusions: Based on the results, anaerobic composting technique could be considered as a feasible, viable and cost-effective alternative for perchlorate bioremediation in the study area. According to the available of techniques and skills, successful experiences of anaerobic composting in other countries, and potential of study area, The application of anaerobic composting is technically feasible and can be use for perchlorate contaminated soil cleanup in a zone war.

Key words: Cow manure, perchlorate, soli bioremediation, technical feasibility

INTRODUCTION

Following occurred wars in the region and extensive use parties involved in the war of thousands tons of missiles and military munitions and explosives containing perchlorates, large areas of residential, agricultural, industrial, and pastures within the border provinces of southern-southwest and west Iran were affected by pollution from of war. The use of explosives contains perchlorates which are used as an oxidizing agent
in solid propellants, missiles, rockets, fireworks, and some munitions in underground applications like mining may result in the release of unused oxidant to environment. \[1\] Khuzestan province due to special geographical and climatic conditions, and the environmental crisis created in the recent years, is at risk types of chemical, biological, and radiological contaminants. Possible sources of perchlorate in surface soils of Khuzestan province, mainly the result of the war(s) occurred in the region, dust storms,\[2,3\] and the excess use of chemical fertilizers.\[4\]

Perchlorates, ionic substances with high melting and very low vapor pressure,\[5\] so does not volatilize from water or soil surface,\[5-7\] in addition solid perchlorates are not expected to directly volatilize to the air as fugitive emissions during their manufacture, processing, transport, disposal, or use.\[8-10\] The perchlorate can precipitate as salts or bioaccumulate, in plant matter.\[1\] Perchlorate does not adhere to soil particles;\[8,9\] however, dissolved perchlorate can be trapped within the soil pores by capillary forces of surface tension (molecular attraction) or becomes trapped in dead-end pore spaces.\[8\] Perchlorate can be retained in some propellant matrices and distributed in soil, and thus not be immediately dissolved.\[1,9\] The presence of perchlorate in surface soil can present potential risks of harm to human health and the environment.\[1\] Perchlorate can enter the food chain and will encounter animal health and humans to serious risk.\[5\] Aquatic and soil environments are possible purpose media for thousands of pollutants such as perchlorate that variable in the compound and in concentration.\[1,8\] Bioremediation is one of the new applications and growing environmental biotechnology. Bioremediation frequently related with contaminated solids such as underground aquifers, soils, and sediments. Microbial processes can be effective in the fate of inorganic environmental pollutants. Detoxification of hazardous inorganic compounds, often naturally through microbial catalysis reactions and these reactions can be strengthened by engineering systems. Anaerobic composting is a controlled biological process in which microorganisms convert perchlorate to fewer harmful byproducts.\[1,7,9]\n
The current technologies to treat contaminated soil include ex situ bioremediation, in situ bioremediation, and ex situ thermal treatment. Anaerobic bioremediation was demonstrated in pilot tests as an effective method for treating perchlorate-contaminated soils.\[11\] Ex situ treatment methods have focused on treatment cells using anaerobic composting or lined treatment cells containing excavated soils. These methods have been evaluated in a pilot- and/or full-scale applications. For composting applications, carbon sources (cow manure, poultry manure, and ethanol) and water, in some cases, bulking agents, are blended with the contaminated soil.\[11\] Efficiency anaerobic bioremediation largely depends on the presence of appropriate perchlorate-reducing bacteria and the ability to stimulate sufficient growth and activity to degrade perchlorate to the extent and rate that meets the intended remedial objectives.\[11\]

Much different substrate can be used to stimulate anaerobic biodegradation. However, the organic substrate(s) selected should be appropriate based on the biogeochemistry and hydrology of the site.\[1,12\] Common substrate types include soluble substrates (lactate, molasses), slow-release substrates (vegetable oil, emulsified vegetable oil, hydrogen release compounds), and solid substrates such as mulch and compost.\[8\] In a study conducted at Aero jet, perchlorate-contaminated soils were prepared in a 7-foot-diameter by 5-foot-high compost pile on an impermeable liner. The soil was mixed with carbon amendments (manure), wetted, and then covered with a plastic tarp to prevent moisture loss and oxygen intrusion. The maximum initial soil perchlorate concentration of 4,200 mg/kg was treated to an average concentration of 0.1-23 mg/kg following 7 days of treatment.\[11\]

A full-scale anaerobic composting was performed at the NWIRP McGregor, Texas, USA. Approximately, 1,500 cubic yards of the perchlorate-contaminated soil were excavated and transported to a biotreatment cell. The soil was mixed with amendments (citric acid, nitrogen, phosphorus, and soda ash as a buffer) and saturated with water. Influent perchlorate concentration in soil was 500 mg/kg. Perchlorate concentrations in the treated soil sampled at six different locations were <100 mg/kg.\[11\]

A pilot-scale anaerobic composting was performed at the Edwards Air Force Base, California, USA. Anaerobic composting of the perchlorate-contaminated soil treated with horse stable compost in 55-gal drums. Initial concentration of perchlorate decreased from 57 mg/kg to the remedial goal of 7.8 mg/kg.\[1,12\]

Anaerobic composting perchlorate-contaminated soil piled 5-feet-high with 7-feet-diameter at the bottom performed in UTC Site, San Jose, California, USA. The average initial concentration of 170 mg/kg was treated to <0.64 mg/kg in <35 days.\[1,12\]

The objective of this research was to investigate the feasibility of the anaerobic bioremediation system for cleanup perchlorate-contaminated soils in a war zone and to design and implement a full-scale anaerobic composting process, in order prevent the release of perchlorate in water and food supplies.

**MATERIALS AND METHODS**

**Study area**

During the Iran-Iraq war, more than 10,000 km² of land area in Khuzestan province were directly under the pollution caused by war. The region under study is a part of a suspected area of contamination that is located west of Khuzestan province, Iran. This region was areas have been affected by war; during the Iran-Iraq war, this region was repeatedly invaded by Iraqi
army [Figure 1]. Based on climate zoning Khuzestan, the study area (perchlorate-contaminated soil), located in the wet zone of low rainfall. This zone, with high humidity and low rainfall, includes the south west Khuzestan province.

The temperature of the warmest and coldest days of the year between 13°C and 34°C fluctuates. This zone with annual rainfall of 167 mm, the lowest rainfall than the other areas. The relative humidity in this zone is 45%. This zone has an area of about 10,920 km², included 17% of the area in Khuzestan province. In Khuzestan province, five major rivers (Karoun, Karkheh, Dez, Jarahi, and Zohreh) with annual discharge of 36 billion cubic meters of water flow. Surface water resources in the study area including the Arvand river (1750 m³/s), and branches of the Karun river. Groundwater investigation showed that there are in Khuzestan province, 3,630 wells, 4 qantas, and 492 springs, and total annual discharge of groundwater resources in the province about 1,075. 31 × 10⁶ m³/year.

In technical evaluation performed for implement a full-scale composting process in the contaminated area, considered a number of questions, criteria, and factors related to successful implementation, including technology and techniques, technical capacity/skills, human and financial resources, constraints or risks to implementation. In selecting appropriate technologies for implementation should exist locally and use before, in addition, skills exist locally to design and implement the project. In technical feasibility studies, constraints of the project implementation any legal, political, or statutory reasons) should be determined.

Composting is an ex situ technique that has been used only infrequently to cleanup perchlorate-contaminated soils. It is a biological process that uses indigenous microorganisms to degrade perchlorate in the presence of organic amendments that support microbial population growth and elevated temperature characteristic of composting. Perchlorate-contaminated soils are excavated and combined with bulking agents and organic amendments such as manure, wood, chips, hay, or agricultural wastes.

Technical feasibility assessment’s cleanup 15 ha of perchlorate-contaminated surface soils of study areas were considered for the implementation full-scale anaerobic composting processes. Accordingly, the number of 15 samples for the determination of perchlorate in the contaminated surface soils were collected. Considering the extent of areas intended for cleanup, facilities needed technically is evaluated.

**Soil sample collection**

Divided the field into the area, so that each sample represents an area of approximately 1 ha. Scrap away the surface litter and insert auger or sampling tube to a plough depth (about 15 cm). Taken at least 10-15 sub-samples with a zigzag pattern to minimize the variability in the sampling area and place them in a clean bucket [Figure 2]. Thoroughly mixed the soil samples taken from 10 to 15 spots from each area in a bucket. By quartering, reduced the bulk and about 500 g of the composite sample is retained. Quartering is done by dividing the thoroughly mixed soil into four equal parts and throw away two opposite quarters. Remixed the remaining two-quarters and again divide it into four parts and reject the two. Repeated this procedure until about 500 g of soil left. Be put the soil into a clean, numbered and stored in polyethylene bags after air drying in the shade at room temperature. Soil samples should be cooled and stored at 4°C ± 2°C, and may be held for a maximum of 28 days before analysis.

![Figure 1](image1.png) **Figure 1:** Map of suspected area of contamination under study in Khuzestan province

![Figure 2](image2.png) **Figure 2:** (a) Sample areas with different properties and sampling zigzag pattern and (b) method of coning and quartering for reducing sample size
Perchlorate extraction from soils
To determine the removal rate of perchlorate of the incubated soils, the extraction method was used in the following steps: For each soil sample analyzed, five 10 g portions were weighed and placed in Erlenmeyer flasks and extracted with 100 ml of solution (Milli-Q [18 MΩ] water) in a tissue homogenizer. Slurry samples were sonicated for 30 min and allowed to cool to room temperature. After extraction and sonication, the samples were centrifuged at 6,000 rev/min for 30 min. Supernatant out of the centrifuged samples was passed through a cartridge of prewashed activated alumina. Samples were filtered with 0.2 µm ion chromatography (IC) syringe filters (Millipore Corporation) and analyzed for perchlorate by IC.[15] Properties of soils examined in this study are in accordance with Table 1.[16]

Chemicals
All chemicals were of analytical grade and were used without further purifications, and inorganic salts used in preparing stock solutions of 1000 mg l−1 perchlorate, were of reagent grade. Sodium perchlorate (ACS reagent, ≥98.0%), procured from Sigma-Aldrich, Germany, was used as the source of ClO₄⁻ in all experiments. All the other chemicals used in this study were purchased from Sigma-Aldrich, Germany.

Machines which are considered for this project include a Loader (Komatsu WAA20), Excavator (Komatsu PC210 LC), Grader (Komatsu GD705 A), Bulldozer (Komatsu D155), Roller (HAMM 5520P), wheel tractor-scraper (Caterpillar 613C), dump truck (Benz 2628), and sprinkler machines which are considered for this project include a Loader (Komatsu WAA20), Excavator (Komatsu PC210 LC), Grader (Komatsu GD705 A), Bulldozer (Komatsu D155), Roller (HAMM 5520P), wheel tractor-scraper (Caterpillar 613C), dump truck (Benz 2628), and sprinkler

Analytical method
A Waters® alliance system IC (Waters Corporation) was used to determine perchlorate. ClO₄⁻ was determined in accordance with EPA method 314.0, with an IC-PAK anion-exchange guard-PAK insert (4.6 mm × 150 mm) and an IC-PAK anion IIC column (4.6 mm × 150 mm). Conditions for the system were as follows: Flow rate, 0.5 ml/min; eluent, 50 mm sodium solution with 18.2 MΩ water; injection volume, 80 µl; ion detection was by waters® 432 conductivity detector; power setting 270 µs/cm, system backpressure, 432 psi; background conductance, 3-4 µs; run time, 25 min.[17] Data from the 432 detector can be collected via Waters SAT/IN™ module and processed with IC-specific parameters by Waters Millennium® Software. Statistical analyses of the experimental data were performed using Microsoft Excel 2013. Waters Corporation is a publicly traded laboratory analytical instrument and software company headquartered in Milford, Massachusetts.

RESULTS
Table 2a shows the concentration of perchlorate in surface soil samples collected from the contaminated area and Table 2b shows the state specific perchlorate advisory levels.

DISCUSSION
In order to prevent surface and groundwater pollution by perchlorate, it needs to keep the water from contacting with the contaminated soil.[10] Since it is difficult to prevent water-soil contact and infiltration into the surface and vadose zone, the treatment of contamination in surface and vadose zone of soils before it reaches to the surface and groundwater is preferable and very important.[1,10]

Based on soil sampling conducted in suspected areas for perchlorate-contamination in Khuzestan province, analysis
of the samples confirmed that contamination [Table 1]. The levels of perchlorate in the soil measured in this study were much higher than the State Missouri advisory levels. Thus, in order to prevent the release of perchlorate into water bodies and food supplies, anaerobic bioremediation, as a cost-effective method, for treating the surface soils was applied in pilot scale.\textsuperscript{[1,14,18]} Iranian department of environment not provided advisory levels for perchlorate in soil.

Before initiating the bioremediation selection process, establishing the cleanup objective is the most important. It is also important to establish the numerical cleanup concentrations, if necessary, and the desired time frame for the cleanup to be complete. A variety of bioremediation technologies are currently commercially available and are being used for cleanup perchlorate-contaminated soil.\textsuperscript{[12]} Within the various sources of projects, machinery, a significant share of costs allocated to them (between 30% and 70%). According to estimate and surveys conducted actual efficiency of machinery in Iran is maximum 50%.\textsuperscript{[19]} Select the type and number of machines for the various works of the project, direct influence on the project implementation time and cost. Factors affecting the selection of machines, including machines efficiency, technical specifications of projects, type of work, volume of work, the technology used to do the work, duration of the project, project budget, and costs.\textsuperscript{[20]}

Locate suitable land for project implementation has been one of the most important steps, and due to the multifaceted nature, is the process very complex. Evaluation criteria for determining a suitable location site, mainly including the slope, topography and soil type, infrastructural services availability in the region, the proportion of dimensions and shape of the site, ownership of land and legal criteria; environmental criteria, status of projects, and development programs. In Khuzestan province, 70 town and industrial zones, including 53 towns and 37 industrial zones with an area of over 8,000 ha. Near study area, two industrial parks with an area of over 2,100 ha are constructed. The main limitations of the implementation project, land mines remaining from the Iran-Iraq war. According to the latest statistic from the UN of countries affected by landmines, Iran is the second country (16 million) in the world with the most landmines. Furthermore, Iraq is most severely affected by landmines and explosive remnants of war as a result of the 1991 gulf war, the 1980-1988 Iraq-Iran war, two decades of internal conflict, and even world war two.\textsuperscript{[21]} Landmines and unexploded ordnance pose a problem in the north, along the Iran-Iraq border, and throughout the central and southern regions of the Iraq.

Considering that project is implementing a large scale, it is essential identifying ownership of the land required for the project, and coordinate with relevant organizations must be done. Since the cleanup of land mine, not performed completely, the lands available in the study area usually Bayer and unused. Land that for the construction of the treatment cell is selected is better flattened. Areas with low unevenness and smooth, in addition to reduce the cost of excavation and initial leveling, the possibility of a systematic treatment of the cells with the appropriate extent provides. Based on data from the slope and topographic and survey maps, the study sites are flat lands.

Based on the results of the treatability studies and a number of other factors, composting was selected as the full-scale remedial action treatment remedy at study area. Appropriate technology selection, design and implementation, for removal and cleanup perchlorate-contaminated soils in under study area, performed based on previous completed projects for cleanup perchlorate-contaminated soils. In order to cleanup perchlorate-contaminated soil using the composting process, a full-scale study conducted in Naval Weapons Industrial Reserve Plant, McGregor, Texas. In this study, perchlorate-contaminated soil was excavated; transported to a bio treatment cell (152 m × 9 m × 1.8 m); mixed with amendments, and placed in the treatment cell lined with 30 mils high-density polyethylene (HDPE). The soil was saturated as it was placed in the cell (0.75 m deep), and more water was added to maintain at least 5 cm of water above the soil to foster anaerobic conditions. The cell was covered with a 6-mil HDPE liner.\textsuperscript{[12]}

To determine the area of land used for the project is necessary that the volume of excavation, filling, and other technical considerations be done [Table 3]. Excavation volume can be calculated using the area of contaminated areas and volume of treatment cells. To perform this project, treatment cell dimension’s study described above is suggested. According to calculation, volume excavation for each cell treatment equal 2,462.5 m$^3$, the volume of contaminated surface soils collected about 22,500 m$^3$, and volume contaminated surface soil was poured into each cell equal 1,026 m$^3$.

In a natural state, the soil is compacted by the pressure of the upper layers, but when excavation is performed, reduced soil compaction and volume increases. Considering this point, the volume of excavation performed must be modified. Assuming a convert coefficient of 0.9 for compacted soils to natural soil, the volume of contaminated surface soils collected about 25,000 m$^3$ and convert coefficient of 1.25 for natural soils to loose soil equal 31,250 m$^3$. The calculations carried out, the number of cells is estimated to be 30 treatment cells. Amount excavation required to build treatment cells and generalization correction coefficients about 102,604 m$^3$. With calculating land required for distribution, the excavated soils of treatment cell thickness of 50 cm, and construction of treatment cells, total area of land required for the project, is about 40 ha.

Estimated cost of excavation, filling, leveling, compression, modification of treatment cell slope, and lined with HDPE liner, per cubic meter of treatment cell is estimated $45. Estimated costs not included costs such as analytical work,
monitoring, sampling, testing, and analysis. In order to cost-effective of the project, the land considered for the project, of government lands in contaminated areas have been selected. Therefore, cooperation and coordination with responsible organizations will be required.

The selection of appropriate organic amendments should be based on a search of local agricultural products and common carbon-based waste streams. Not all potential amendments will be locally available or be the most cost-effective option in different areas of the country.[1,11] The selection of the most appropriate amendment should be site-specific and is typically based on the degradation rates measured in microcosm studies. However, several other issues must be considered in the selection of an amendment, including cost, supply, presence of impurities or nuisance compounds (e.g., sulfur in molasses), and the ability to obtain permission for injection of the substance into the subsurface. Another consideration is whether a soluble amendment (e.g., acetate) is appropriate or an insoluble amendment (e.g., vegetable oil).[1]

The selection of co-substrates (ethanol, cow and poultry manure) for present study as carbon sources, conducted based on several considerations that include: (a) Literature study in order to identify a suitable organic substrate according to the research conducted in this field, (b) field visit to assess and identify a variety of organic substrates in contaminated area and its surroundings, (c) costs of organic substrate (local availability, cost of substrate, size of area to be treated (quantity needed), depth of incorporation (application rate/quantity needed), transportation costs), (d) substrate longevity, (e) form of application, (f) soil texture, and (g) desired results.[1,10,11]

The pilot in situ bioremediation was conducted at a longhorn army ammunition plant, Texas by Nzengung, et al. Based on the results of batch and laboratory column tests that evaluated the ability of different organic amendments to stimulate perchlorate degradation by naturally occurring bacteria. The tested organic amendments included cow manure, chicken manure, methanol, ethanol, acetate, molasses, and cotton gin waste.[15]

As shown in Table 4, according to the census conducted by the statistical center of Iran of dairies industrial, broiler and laying poultry farming in 2012, there are 531 unit’s dairies industrial, 742 units of broiler farming, and 6 units laying farm in Khuzestan province. The annual productions of manure were 32,100 tons (dry manure) from dairies industrial, 47,234 tons of broiler farming, and 2,621 tons of laying farming. The price per ton of cow manures (dry) $6.587, broiler manure $20.522, and lying manure $10.965 was estimated. Price per cubic meter of ethanol 96%, equivalent to $1,748.[23]

Organic substrates that are rapidly depleted require more frequent injection to develop and sustain sufficiently reducing conditions. Form of application solid substrates (cow and poultry manure) used for anaerobic bioremediation is trenches, excavations, or surface amendments.[1,10,11] The typical injection frequency is one-time emplacement and the typical life span is unknown, but thought to be 5 years or more.[1] Based on previous studies (Table 1), the soil texture of the study area is composed of 23.26% sand, 50% silt, and 26.68% clay.[16]

Based on the literature study and field visit of the contaminated area under study, selected three types of organic substrate for treating perchlorate-contaminated soils.[1,24] Local availability is the most important consideration in the selection of substrate. Due to the high amount of poultry manure produced in the Khuzestan province, compared with cow manure and ethanol is more cost-effective. Choosing manure as substrate, compared with poultry manure and ethanol is less expensive. The typical injection frequency in solid substrate (cow and poultry manure) is one-time emplacement and the typical life span is being 5 years or more, while soluble substrates like ethanol depleted relatively quickly.[1] To evaluation of important technical factors in selecting an anaerobic bioremediation process for cleanup contaminated soil, and evaluation technical feasibility.

Table 3: Activities necessary to construction treatment cell[22]

| Construction treatment cell activities | Iran rials (IRR) | USD ($) |
|--------------------------------------|----------------|---------|
| Plough with mechanical devices, to a depth of 15 cm | 300/m² | 120.65/ha |
| Excavation in soft ground and transporting material resulting from excavation to distance 20 m of the excavation, and masses it | 4130/m³ | 166.64/1000 m³ |
| Channel digging to a depth of 2 m in the land of soft by mechanical device | 14,500/m³ | 585.47/1000 m³ |
| Downloading material from earthworks or soil mass and transported by truck to distance 500 m | 14,000/m³ | 564.7/1000 m³ |
| Flattened levee bed by grader | 260/m² | 104.83/ha |
| Sprinkling and banging the floor of trenches up to a depth 15 cm | 1060/m² | 427.88/ha |
| Mixing materials together | 3510/m³ | 141.42/1000 m³ |
| Distribute materials from excavation, with each thickness | 1680/m³ | 67.745/1000 m³ |
| Create route surface water collection into waterways | 450.8/m | 18.2/1000 m |
| Create of surface water collection channels | 565/m | 22.746/1000 m |

*1 USD = 24,773 IRR January 1, 2014 According reports Central Bank of the Islamic Republic of Iran
assessment, ability to execute process, technically there in the contaminated area.

CONCLUSIONS

The purpose of this study was to assess the feasibility of an anaerobic composting process for perchlorate-contaminated soils in study area. Considering perchlorate-contamination surface soils in the study area and the urgent need to clean up the soil contamination to prevent the release of perchlorate in water resource were conducted technical feasibility study for cleanup of soils contaminated with perchlorate. To assess the feasibility, a complete study of questions, criteria, and factors related to successful implementation, including technology and techniques, technical capacity/skills, human and financial resources, constraints or risk to implementation characterization was done. Then three common treatment options were considered for cleanup perchlorate-contaminated soil in study area. It was determined that anaerobic composting was the most feasible option based on the technical, climate, and logistical issues on study area. It was shown that anaerobic composting is a solid project economically, environmentally, and educationally. Considering all the technical factors affecting the process of cleanup, was found that the large-scale cleanup of perchlorate from contaminated soils in the study area can be done.

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