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Volume II of II

SOLVENT EXTRACTION OF FLARE PIT SLUDGE USING A SOIL AGGLOMERATION APPROACH

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INTRODUCTION

A large number of remediation technologies, for the removal of a variety of contaminants, are currently being investigated. Although some of these processes work well in specific cases, nearly all have limitation with respect to costs, contaminant type and soil properties. The problems associated with these technologies are further exacerbated since landfarming and landfilling are being reduced or abandoned because of environmental concerns. Solvent and aqueous extraction or washing processes have been shown to be effective for treating many of these contaminated soils, sludges and sediments.¹ However, both extraction methods only work well on coarse material. Treatment of fine textured soils is less effective as intractable fine particulate dispersions are often formed in the liquid extraction medium.

Prior to the mid 1980's most oil and gas wells, gas plants and refineries had some form of flare pit, usually an excavated area at some distance from the installation, fitted to allow transport and burning of gas or liquid petroleum. Some of these pits were designed for emergency situations while others were intended to destroy uneconomical or waste organic materials. The soil surrounding the pit often contained a wide spectrum of organic material, ranging from highly volatile organics to partially combusted heavy oils and coke-like materials. To compound the problem, flare pits often became convenient burn pits for the disposal of tank bottoms and other various and sundry waste products. Also, berms built around these pits were frequently sprayed with chemical defoliant. In Canada, flare pits now require linings and open pit disposal is being discouraged but the legacy of tens of thousands of these pits with associated organic contaminated soil and partially burnt sludge remains.

SESR PROCESS

A generalized solvent extraction block diagram is shown in figure 1. It consists of a mixing and contacting devise to transfer the contaminant from the solids to the solvent. A solids-liquid separation step is next followed by the recovery of residual solvent from the solids, usually by thermal drying. The solvent is separated from the contaminant and recycled back to the extractor. Solids liquid separation is the critical step for most solvent extraction

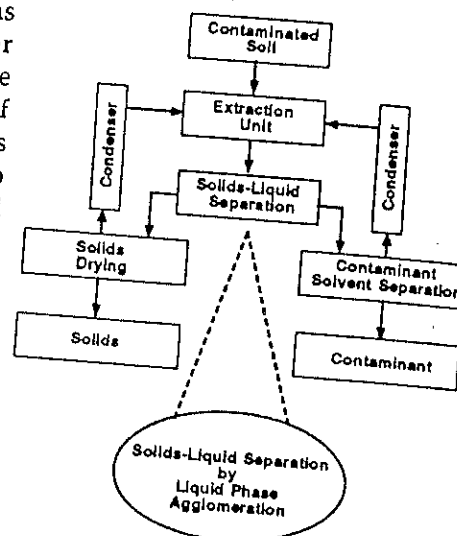


Fig 1 General solvent extraction flow diagram

processes, especially when large quantities of fines (-50μ) are involved. Commercial success depends on how these non-settling fine solids are handled as they have a tendency to plug percolation beds or blind filters.

The National Research Council of Canada has developed a solvent extraction process, SESR (Solvent Extraction Soil Remediation), in which the fine and coarse particles are bound together using a liquid phase agglomeration technique.² This approach utilizes a size enlargement technique that improves the separation of fine solids from either aqueous or organic based suspensions or slurries. A second liquid must be selected that will preferentially wet, or absorb on the surfaces of the particulate solids, while being virtually immiscible with the slurry suspending medium. Addition of a minor amount of this second, or bridging liquid to the solids slurry, in conjunction with suitable agitation, will result in attachment of primary particles to form aggregates. The strength of the bonding between particles results from the capillary forces arising after interaction between adsorbed layers of bridging liquid during particle collisions in the agitated slurry/suspension. The form and size of the agglomerates produced are primarily dependent on the size and type of particles, the amount of bridging liquid added and the degree and type of agitation applied. A complete description of the process is found elsewhere.³

MATERIALS

Flare Pit Sludge:

A sample of flare pit sludge material was supplied by the Alberta Environmental Centre and was taken from a pit associated with an oil well. This well produced uneconomical solution gas which required flaring. The oil was considered sweet and hence the lack of sulfur compounds found with the organic contaminants. The pit also contained a significant amount of wax which was thought to have been present as a result of the disposal of pipeline de-waxing pigs.

Contaminant analysis was carried out by exhaustive extraction with toluene in Soxhlet units fitted with Dean and Stark water separators. Results from 24 feed sub-samples gave the following average composition: $50.9 \pm 0.8\%$ w/w solids, $28.7 \pm 1\%$ w/w water and $20.5 \pm 1.4\%$ w/w toluene solubles, by difference. The organic material had an initial boiling point of $<50^\circ\text{C}$; infra-red and NMR spectra of the extract showed it to be mainly aliphatic in nature. Simulated distillation gas chromatography showed over 320 different components with boiling points below 542°C , the limit of the apparatus. Vacuum distillation at 90°C to remove all light ends followed by Soxhlet extraction with methylene chloride and subsequent evaporation of the methylene chloride gave a slightly lower organic content with 7.3% being in the lighter vacuum fraction. Using a Leco CR-12 Carbon analyzer, total and residual organic carbon contents were obtained and extractable hydrocarbon calculated.

Erskine Soil:

A contaminated black, Chernozem soil from near Erskine Alberta along with an uncontaminated sample from an adjacent site was used in these trials. Both were collected from surface soil horizons. The soil had been contaminated with a mixture of brine and crude oil from a pipeline break between the well-site and the battery. Analysis showed a hydrocarbon content of 5.6% with a high asphaltene level. The presence of brine resulted in a pH of 7.7 and an electrical conductivity of 26.8 mS/cm. The extreme water repellent condition of this soil was indicated by the fact that a 9.2N ethanol solution was needed to penetrate it in the dry state.

Other Materials:

Invert Drilling Muds contaminated with diesel fuel, PCB contaminated soil from a transformer oil spill and waste material from a decommissioned plant that had manufactured 2,4,5-T herbicides; (2-methyl-4-chlorophenoxy) herbicides; 2,4-D herbicides and wood preservatives, primarily pentachlorophenol, have also been investigated.

METHODS

In the current work, a series of batch scale experiments were conducted using organic solvents as the extracting liquid and water as the bridging liquid. Three types of agitation were employed; (1) a complex reciprocating action (paint shaker), (2) high speed mixers (blender) and (3) tumbling at 20% of the critical speed in a rotating cylindrical mill containing mild steel rods. At a residence time of 5 minutes, the latter closely simulates a full scale process and was used for most work. As the agglomeration process is water-limited it may be necessary to either increase or decrease the water content. This can be achieved by direct addition of water or by adding dryer soil or an additive like peat moss. During processing, solvent extraction of the contaminants occurred concurrently with disaggregation and subsequent agglomeration of the dispersed soil particles. The agglomerates were usually surfaced washed with fresh solvent and air dried at 110°C before being analyzed.

Four solvents with different polarities were selected; paraffinic (heptane); aromatic (toluene); chlorinated (trichloroethylene) and commercial naphtha (Varsol®). The Hildebrand solubility parameter (δ) was used to characterize each solvent.⁴ Other parameters investigated included pulp density, degree of agitation, amount of bridging liquid and pre-mixing of solvent. Interactions were examined by determining relative mass transfer coefficients⁶ calculated from data on the rate of transfer of contaminant into the solvent.

RESULTS AND DISCUSSION

Figure 2 shows the recovery of contaminants with respect to a variety of solvents. In this case we have plotted the recovery as a function of solubility parameter (δ) where:

$$\delta = \gamma(M_w/\rho)^{1/3}$$

Flare pit sludges show no major differences with respect to the solvents tested. This is not unexpected given the aliphatic nature of the contaminant. Consequently, a solvent with low environmental impact such as heptane can be selected. In the case of the Erskine soil there is a significant solvent effect. This can be attributed to the high molecular weight asphaltene content of the crude which is only sparingly soluble in paraffinic solvents but is readily dissolved by chlorinated and aromatic solvents.⁵ Remediation of the other described contaminated samples gave similar results with removal of approximately 95% of the offending material.

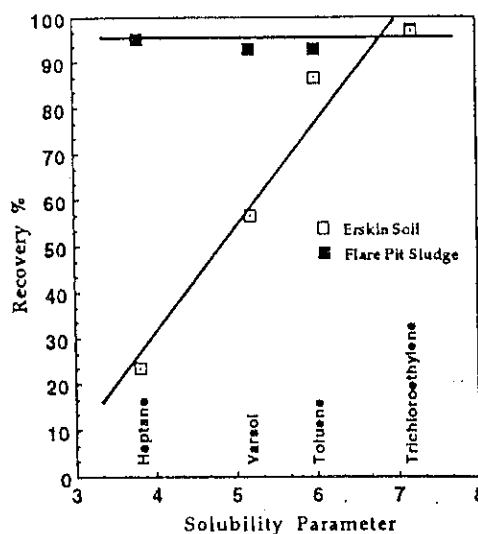


Figure 2 Recovery of Contaminants

Figure 3 illustrates the use of the relative mass transfer coefficient K_a/K_s to determine the interaction of operating parameters on the rate of extraction⁶, where subscript s represents a standard set of conditions and subscript a, the comparative set. The effect of pulp-density, defined as the percent of dry solids in the slurry in the extraction agglomeration mill, is illustrative of this type of analysis where we show that an improvement in extraction is obtained by increasing pulp density. We attribute this change to increased abrasion effects due to more particle-particle interaction. This technique shows that addition of mixing rods, extraction temperature, solvent pre-loading with contaminant and rotational speed are all parameters which affect extraction rates.

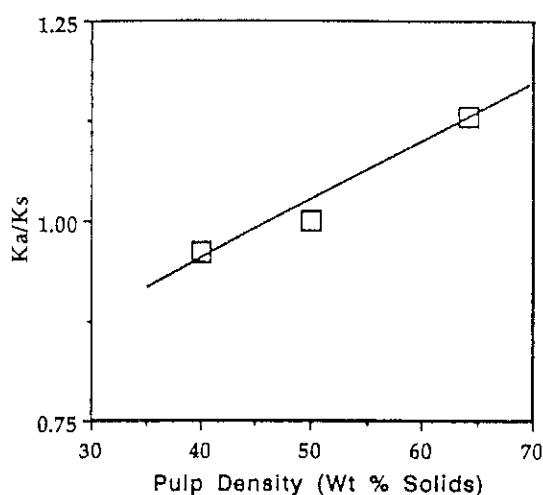


Figure 3: Relative Mass Transfer Coefficient as a Function of Pulp Density

CONCLUSIONS

It has been demonstrated that fine textured soil can be remediated and that relative mass transfer coefficients can be calculated to determine interactions and critical parameters.

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