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#### **APPROVALS**

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The SESR Process: Remediation of Soils, Sediments and Sludges

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### Oil Sands Research at the National Research Council

BD Sparks, September 1994

NRC, first through the Division of Chemistry and more recently the Institute for Environmental Research and Technology(IERT), has been involved in oil sands research and development projects for about 40 years. In the early "fifties" Peterson and Gishler studied an oil sands retorting process using fluid bed technology. Energy requirements for the process were supplied by combustion of the coke deposited on sand grains during thermal cracking of bitumen. Later, in 1960, a cold water, bitumen agglomeration approach was used as a separation technique by Puddington, Meadus et al. This work was carried out at the same time, but independently of, the similar sand reduction approach taken by Imperial Oil. A continuous extraction device, based on this principle, was later developed and tested at NRC during the period 1965-1969 (Sparks & Meadus). Sun Oil Co., the parent company of GCOS, evaluated the unit for recovery of bitumen from low grade ore and sludge, using partial bitumen product recycle to maintain efficient operation.

Prompted by the unexpectedly large sludge volume during start-up of its HWE plant in 1967-1972, GCOS began looking at alternative bitumen extraction processes. The capability of treating low grade ores and minimizing sludge production were considered to be the most desirable feature of such technologies. Solvent extraction totally avoids the formation of HWE type sludges. However, the clean separation of bitumen solution from finely divided solids is itself a difficult problem. Use of solids agglomeration techniques largely overcomes this drawback and the combination of Solvent Extraction and Sand Agglomeration led to the development of the SESA process (Meadus & Sparks) which was licensed by Terra Energy Ltd. (TEL) in 1974.

In its original form SESA used a pelletizing process to desolventise mechanically the extracted sand by water displacement. This approach could be used for high fines, i.e. usually low grade ore. A technical assessment and economic evaluation by MHG International Ltd., Calgary, in 1978-1980 indicated that the limitation to high fines feed precluded use of the process for stand-alone operation in the Athabasca oil sands region. At the time of the Canstar project, Petro-Canada evaluated a number of alternative extraction schemes and selected the SESA process as the best available solvent extraction method. Process modifications were made to allow treatment of all grades of oil sands and a small, integrated pilot plant was tested at the Petro-Canada facility in Calgary. The project was jointly funded by NRC, through the IRAP programme, and Petro-Canada.

This successful process demonstration led TEL to apply to the Alberta Oil Sands Technology and Research Authority (AOSTRA) for funding to design a Demonstration Plant that would be build in the Fort McMurray area. In 1988 a contract was awarded to Kilborn Energy Inc. to provide:

- · size selection and conceptual design of the demonstration plant;
- · an economic evaluation and comparison of SESA and HWE; and
- a detailed design and costing of the demonstration plant.

The economic assessment showed that the SESA process could be compared favorably to the HWE process when the cost of final tailings handling and disposal was included in the estimates.

In addition to development of the alternative extraction processes, NRC also is involved in fundamental studies of HWE tailings sludge stability as part of the Sludge Fundamentals Consortium, a research program involving ARC, NRC, Suncor, Syncrude, AOSTRA, CANMET, Environment Canada and Alberta Energy. The ultimate objective of this consortium is to develop viable tratment technoloiges for existing sludge and/or process modifications to prevent formation of additional sludge.

During years of research on oil sands, the Process Technology group at IERT/NRC has built up considerable expertise in this field. Numerous papers have been published and patents have been issued on all aspects of research and process development for oil sands extraction and related environmental issues, including the remediation of organic-contaminated soils.

#### THE SESR PROCESS FOR SOIL REMEDIATION

Background

Many soil remediation technologies have been described in the literature. A summary of ex-situ technologies is given in Table 1 (1). In terms of cost, land filling is the bench mark for remediation technologies, however, this option does not separate or destroy contaminants and is becoming less available or acceptable. Incineration of contaminated soil is expensive and may produce a hazardous residue through transformation of heavy metals into leachable forms. Other processes also have some limitations. In particular, a high clay or silt content in the soil is quite restrictive for many applications, especially soil washing. High concentrations, or toxicity, of contaminants have

adverse effects on most biological treatments. Also, contaminated soils are often associated with complex mixtures of pollutants which may require a train of different technologies for satisfactory remediation. Consideration of these factors indicates the need for a technology capable of handling fine textured soils, contaminated with high concentrations of oils or toxic compounds. Such a technology should be compatible with other processes so that it can be readily incorporated into an integrated remediation approach. Overall costs should be less than that for incineration of the untreated soil.

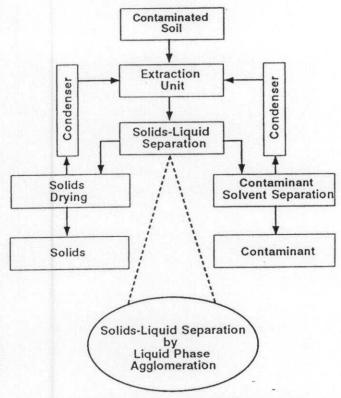


Figure 1: Generic Solvent Extraction

Solvent extraction (2) has been wide-

ly investigated because of its potential for treating a broad range of difficult to treat contaminants at relatively low cost. While a number of solvent extraction processes have been commercialized these existing technologies are restricted to applications for coarser soils. Although contaminants are not eliminated the volume of material for final disposal is much reduced, thereby producing overall cost benefits.

Figure 1 shows a simplified diagram of a generic solvent extraction process. The basic unit operations include; a mixing stage to promote solvent-contaminant contact, a solid-liquid separation step and means to recover solvent from both extracted solids and contaminant solution. Recovery of solvent for recycle to the process is an important feature from both an economical and environmental point of view. In this treatment scheme fine textured soils present a problem because process effectiveness ultimately rests on the need to achieve efficient solid-liquid separation. For example, while contaminants may be readily dissolved by a good solvent the overall effectiveness of treatment is still governed by the ability to separate the resulting solution from the extracted soil. The operation of solid-liquid separation units is primarily governed by the particle size distribution of the solids being treated. As particles become finer their surface area increases while the pore spaces between them become smaller. These factors produce large surface tension effects which act to prevent clean separation of the solvent phase from the fine solids.

#### Liquid Phase Agglomeration

The National Research Council has developed several versatile technologies based on Liquid Phase Agglomeration (LPA) (3,4). One application is to improve the separation of fine solids from liquid suspensions by particle agglomeration. LPA is a size enlargement process based on agglomeration of dispersed solids in the presence of two largely immiscible liquids, one of which will preferentially wet the particle surfaces. The wetting, or bridging liquid, is usually present in relatively small amounts, ranging from 10 to 50 w/w % of the solids. The major liquid component, the suspending medium, may comprise 30 to 80 w/w % of the solids slurry. To initiate solids' agglomeration, sufficient agitation must be used to disperse particles and bridging liquid throughout the suspending medium. During this mixing stage fine droplets of bridging liquid are contacted and adsorbed by the dispersed solids. Agglomeration occurs as a result of bonds formed between particles through bridging liquid layers during particle collisions in the slurry. The strength of these bonds results from capillary forces within the bridging liquid at particle junction points; finer particles produce greater bridging liquid surface curvature and hence stronger agglomerates. Another important criterion is the wettability of the solids by the bridging liquid; best results are obtained when the solid surfaces are completely compatible with the bridging liquid. If necessary surface conditioning agents may be used for surface modification.

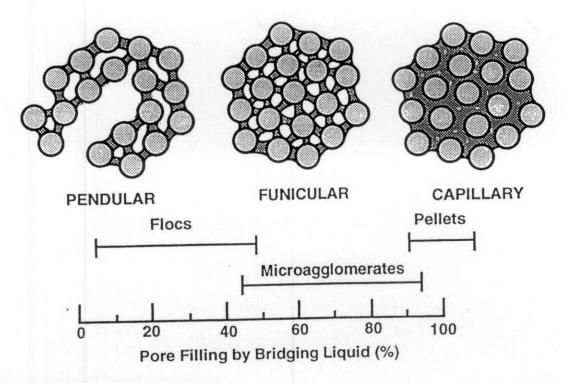


Figure 2: Agglomerate Forms

Figure 2 summarizes the agglomerate characteristics associated with different amounts of bridging liquid; low levels give incomplete coverage of particle surfaces. The limited number of pendular bonding points, on each particle, results in the formation of open structured flocs that entrap large amounts of the suspending medium. If the latter contains dissolved material this condition leads to poor separation of the solute. Increasing the amount of bridging liquid eventually produces a continuous, adsorbed, liquid layer around each wettable particle; the system is then in the funicular state. There is now no limit to the number of particle bonding interactions and the flocculated structure will condense to form more compact agglomerates. The combined effect of capillary forces and agitation produces roughly spherical bodies. Although the bridging liquid now forms a continuous network throughout each agglomerate, its volume may still be insufficient to completely fill the internal porosity. Retention of suspending liquid in the intra-agglomerate pores may remain high, again leading to unacceptable levels of retained solute. When the agglomerate pores are saturated with bridging liquid the system is in the capillary state. Under these conditions the agglomerates achieve a maximum in density and strength. Optimization, by control of agitation and amount of bridging liquid, allows the suspending medium to be virtually eliminated from the internal pore volume of the agglomerates. If the original soil is oversaturated with water it may not be possible to form agglomerates without some pretreatment such as drying, addition of unsaturated soil or blending with suitable amendments such as peat, lime or gypsum.

Solvent extraction, of fine textured soils or sediments, provides an almost ideal application for LPA. The suspending liquid can be selected to be a good solvent for the contaminant and water may be used as the bridging liquid for hydrophilic soil particles. One restriction is that the solvent should be virtually immiscible with water. In some instances contaminants may be strongly bound to soil particles; this usually occurs with the finest size fractions and in such cases the particles may be rendered hydrophobic. The surface selective nature of the agglomeration process results in the beneficial rejection of these contaminated, hydrophobic particles from the agglomerates. The unagglomerated solids remain with the solvent phase and are ultimately concentrated with the contaminant for final disposal. This capability for separation of a minor hydrophobic solids fraction, while agglomerating the remaining hydrophilic particles, to improve solid-liquid separation, is a unique feature of LPA technology. The combination of contaminant extraction with solids agglomeration has been designated Solvent Extraction for Soil Remediation, or the SESR process.

**Process Description** 

Figure 3 shows a conceptual flow sheet for a multi-stage SESR plant. In the primary stage the soil or sludge, solvent and water are slurried together in a tank agitated by a turbine mixer. Amendments, for moisture control, or metal fixation may be added at this stage. The contaminant is dissolved while coarse and fine soil particles are agglomerated through capillary bonds formed between adsorbed layers of the water phase. The strength of these bonds is governed by interfacial tension, particle size and the contact angle of water with respect to the solids. Control of moisture content and degree of agitation allows the solids in the slurry to be formed into spheroidal agglomerates having a desired, specific size distribution. Any hydrophobic solid particles will tend to remain dispersed in the solvent phase.

The output from the primary extraction step passes through a solid-liquid separation device (cyclone, screen or screw classifier) to the next mixing stages. Solvent flow is set up to be counter current with the agglomerated soil solids so that, in each stage, the solids are contacted with progressively cleaner solvent. The mixing conditions in each tank must be adjusted so that an equilibrium between breakdown and re-formation of the soil agglomerates is established to reproduce the desired size distribution. Breakdown of the agglomerates in each mixing vessel is necessary to allow the small amount of dirty solvent, trapped in agglomerate pores, to be replaced by the cleaner solvent in the current extraction stage; surface washing of the solids occurs at the same

time. The number of stages required depends on the effectiveness of the solid-liquid separation method, solvent-solids contacting, the amount of contaminant dissolved and its concentration at each step.

Depending on agglomerate size, additional mechanical deliquoring of the agglomerates may be advisable after the last extraction step; this may be accomplished by filtration or centrifugation. Residual solvent and water are then removed from the agglomerated solids in an externally heated, rotary dryer with a blanketing atmosphere of steam. During this drying step any water soluble contaminants are brought to the agglomerate surfaces through efflorescence effects; this is an important feature if, for example, a leaching step is required to remove high concentrations of salts or dissolved heavy metals. The contaminant solution must be concentrated by distillation to recover the bulk of its solvent content for recycle to the process. For high molecular weight contaminants a combination of membrane concentration and distillation may be adopted to optimize energy consumption.

Figure 4 is a detailed flow sheet for a 2 tph SESR demonstration plant with four extraction stages. The first stage is a rotating mill containing rods and the succeeding stages incorporate stirred tanks. The use of a rotating device allows oversized material to be removed through a trommel screen. Solid-liquid separation is accomplished by a screw classifier, after the rotating mill and by a cyclone after each stirred tank. The residual solvent is recovered from the agglomerates using an externally heated rotary dryer with a steam sweep. The solvent is separated from the contaminant using conventional distillation.

#### Advantages of the Technology

Evaluation of conventional solvent extraction processes has shown that they are:

- effective for treatment of sediments, sludges and soils containing organic contaminants such as polychlorinated bipheyls, other chlorinated compounds and petroleum wastes
- contaminants are concentrated to a small volume for final disposal
- fugitive emissions and other solvent losses amount to 2-5 L/t of solids treated
- · unit operations use standard equipment
- processes are amenable to modular construction for ease of transport and erection

These processes have limited applicability in the presence of silt or clay because solid-liquid separation is an inverse function of solids particle size. However, a combination of solvent extraction and agglomeration technologies provides the following additional benefits:

- · much improved solid-liquid separation for fine textured soils and sediments
- · stronger, more stable agglomerates at high clay and silt concentrations
- · decreased dust formation during handling and transport of treated soil
- organic coated fines may be selectively rejected from the agglomerates and concentrated with the contaminant

Agglomerated soils and sediments provide the following advantages when used in combination with other treatments:

- closely sized stable agglomerates are ideally suited for post treatment leaching e.g. for brine and heavy metal removal
- amendments may be incorporated during agglomeration to promote bioremediation or to fix heavy metals
- agglomerates emulate natural soil aggregates in terms of aeration and drainage properties
- in some circumstances natural micro-organisms may be re-established by inoculation with clean top soil - the resulting medium will support plant growth

Unit operations have been extensively tested at a small pilot plant scale. Similar agglomeration units have ben commercialized for other applications. e.g. coal washery waste treatment. The application of this process allows treatment of fine textured soils, or sediments, heavily contaminated with non-volatile or toxic materials.

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