Invert Mud Encapsulation Using Pozzolans and Portland Cement

1. Introduction

- The purpose of this collection of literature is to demonstrate the efficacy of pozzolan-modified concrete for stabilization, fixation and encapsulation of drilling wastes, particularly oil-based muds. While not commonly practiced in Alberta and not addressed in AEUB Guide 50 "Drilling Waste Management", there is considerable operational experience elsewhere in the world to suggest encapsulation using a pozzolan and Portland cement admixture as an Alternate Method within the meaning of Guide 50.

- Drilling wastes consist of drill cuttings, produced formation fluids and drilling fluids. These wastes may be quite complex, depending upon the drilling, hole completion, formation testing and stimulation history of individual wells. Encapsulation is primarily concerned with the disposal of spent oil-based muds, although it may have application to other categories of waste as well, particularly those where fixation of heavy metals and trace elements is involved.

- There are six commonly used solidification agents: Portland cement, pozzolans, bentonite, lime, plaster of Paris, thermoplastic resins and other polymers and ion exchange resins (zeolites). Of these, the most important agents for waste fixation are Portland cement, pozzolans, bentonite and zeolite.

- Portland cement is a synthetic mineral formed from calcium silicate. When mixed with water, it forms a combination of hydrated calcium silicate and Portlandite (hydrated calcium hydroxide). Young (1992) discusses the key properties of cement-based materials for waste solidification and encapsulation. The key properties are: formation of a strong solid matrix; a suitable pore structure for storage of materials within the matrix, which can be controlled by additives; and an intrinsically high pH for fixation of metals. Young (1992) also describes the effects of pozzolanic additives on permeability and suggests measures to reduce the brittleness of Densified Small Particle (DSP) concrete. The paper describes several methods for offsetting the negative effects of increased brittleness.

  The long-term effects of encapsulation of hazardous wastes in cement have been an important topic for research for much of the past 20 years (Newman, 1992), driven largely by the need for quality assurance of treatments for high level nuclear materials.

- The Dictionary of Geological Terms (American Geological Institute, Second Edition, 1962) defines Pozzolan as: "A leucitic tuff quarried near Pozzuoli, Italy, and used in the production of hydraulic cement. The term is applied more generally to a number of natural and manufactured materials (ash, slag, etc.) which impart specific properties to cement. Pozzuolanic cements have superior strength at a late age and are resistant to saline and acidic solutions." Gibbons (undated), in an article posted on the UK Building Conservation website (www.buldingconservaiton.com/articles/lime/pozzo.htm) describes the properties of pozzolans and their beneficial effects on cement and mortars. The State of Virginia, USA describes how it uses blast furnace slag together with pozzolans to make durable concrete for public works purposes (www.vdot.state.va.us/btrc/briefs/00-rlrb/Hydraulic_Concrete_Strategies.htm).

- Zeolites are a natural metamorphic rock type produced by the hydrothermal alteration of lava, volcanic ash and related volcanic debris. Zeolites are characterized as having an aluminosilicate framework whose structure contains channels filled with water and exchangeable cations; ion exchange is possible at low temperature (100 C at most) and water is lost at about 250 C. Water (and other mobile substances) is re-adsorbed at room temperature (Gottardi and Galli, 1985).

  Synthetic zeolites, notably Zeolite A produced by Union Carbide in the late 1940's, are also well known (Dyer, 1988). The synthetic zeolites have the same open structure as the naturally occurring species and consequently the properties of ion exchange and reversible water loss, but have a wider range of chemical composition. Germanium, gallium, beryllium and phosphorus can substitute for silicon in the Primary Building Unit (PBU), the silica tetrahedron. Synthetic zeolites are sometimes referred to as 'zeotypes" to differentiate them from naturally occurring metamorphic rocks.

- Fogg and Berzins (1993) describe the chemistry of cement as it applies to the containment of hazardous substances, noting the shortcomings of the method for fixation of Volatile Organic Compounds (VOC). They also present a chart showing the effects, both positive and negative, of a range of substances on the integrity of cement-based pozzolanic processes. As an appendix to the paper, Fogg and Berzins note that British Columbia has adopted the Environment Canada document "Proposed Evaluation Protocol for Cement-based Solidified Waste, EPS 3/MA/9".

- The "Proposed Evaluation Protocol for Cement-based Solidified Wastes" (Stegemann, 1991) is a three level procedure for evaluation of the efficacy of fixing inorganic waste residues. Level 0 in the protocol provides basic information about the process and identifies contaminants to be monitored in the leaching tests. The organic content of the matrix is the only performance indicator for which a performance criterion must be set. Methods for achieving this goal are appended to the text.

  Level 1 of the evaluation protocol partitions the waste matrix into three components based on leaching potential:

  1. the amount readily soluble (or the initial leachate concentration), which represents the short-term potential for contaminant leakage;
  2. the amount not readily soluble but available for leaching, representing the intermediate term potential for leakage;
  3. the amount unavailable for leaching, representing the long term potential for leakage.

  Methods for assessing the performance of the stabilized material against performance criteria are included in the text of the Protocol.

  Level 2 of the Protocol evaluates the degree of physical entrapment of contaminants in a monolithic specimen. Testing at this level includes dynamic leaching (indicates the rate of diffusion of constituents out of the monolith), permeability testing, durability testing (which includes physical, chemical and biological mechanisms of attack); wet-dry and freeze-thaw cyclic testing (resistance to weathering); and demonstration of the inability of the monolith to support life in any form. Specific testing methods are prescribed for each category of attack.

  Criteria for performance have been developed from Environment Canada and USEPA databases on a wide range of contaminants and stabilization-solidification technologies.

2.

Characterization of Invert and Oil-based Muds

2.1. Chemistry of Drilling Fluids

- Gray and Darley (1980) classify drilling fluids on the basis of their principal fluid components, that is water, oil and gas. Table 2.1 (derived from Gray and Darley, 1980) summarizes the general types of drilling fluids currently in use in the world petroleum industry. In most cases, a solid phase exists in the drilling fluid, conferring the title of "mud" to the fluid. A suspension of solids in water is referred to as a "water based mud", while a suspension of solids in oil is an "oil-based mud".

TABLE 2-1: CLASSIFCATION OF DRILLING FLUIDS
ACCORDING TO PRINCIPAL CONSTITUENT

Gas	Water	Oil
Dry gas: Air, natural gas, exhaust gas, combustion gas	**Fresh water:** no additives	**Oil:** Diesel or crude
Mist: Droplets of water or mud carried in the air stream	**Solution:** True and colloidal, i.e., solids do not separate from water on prolonged standing. Solids in solution with water include: • Salts (NaCl, KCl, CaCl • Surfactants (detergents, flocculants, coagulants) • Organic colloids (cellulose and acrylic polymers)	**Oil Mud:** A stable oil-base drilling fluid contains: 1. Water-emulsifying agents 2. Suspending agents 3. Filtration-control agents Contains cuttings from the formation drilled. May contain barite to raise density.
Foam: Air bubbles surrounded by a film of water containing foam-stabilizing surfactant	**Emulsion:** An oily liquid maintained in small droplets in water by an emulsifying agent (diesel oil and a film stabilizing surfactant)	
Stable Foam: Foam containing film-strengthening materials, such as organic polymers and bentonite	**Mud:** A suspension of solids (clays, small cuttings) in any of the above liquids, with chemical additives as required to modify properties	

- When both oil and water is present, the fluid is called an emulsion, which may be accompanied by both solids and an emulsifying agent. When oil dominates, the fluid will be called an "oil emulsion mud" and if water dominates, the fluid will become an "invert emulsion". All spent drilling fluids will contain some water, however those muds that have intentionally included water as a constituent will not usually separate by itself over time without some kind of preliminary treatment.

- The solid phase generally includes thickening and suspension agents, as well as barite. The emulsified water may also carry alkalis and salts in solution.

- A variety of materials are added to drilling fluids in order to modify their physical and chemical behaviour. The most common additives are bentonite clay, diesel fuel, brines, starch, cellulosic polymers, lime, chrome-lignosulfonate, lignite, sodium chromate, surfactants, weathered crude oil, asphaltic crude, asphalt, various emulsifiers and acrylic. In addition to these primary additives, spent drilling fluid will also contain rock chips and fluids derived from the formations penetrated by the bit. The primary materials may be available from the rig mud log, although proprietary formulations added to the drilling fluid may not be well documented, hence the need for assaying before treatment and disposal may be attempted.

- Macyk et al (1992) have extensively sampled spent drilling fluids from sumps all over Alberta. Their results indicate that 74% of wells produces 1,000 m^3 or less of spent mud, which is composed of roughly equal volumes of solids and liquids. Wells west of the 5th Meridian leave about 30% more mud than those to the east, reflecting the deeper targets explored along the disturbed belt. Gel muds are the most common (58%), and invert the least (12.3%). Only one sump containing brine drilling fluid was found of the 106 well sites evaluated. Liquid phase waste chemical parameter magnitudes that were judged as being of concern were electrical conductivity (EC), chloride ion (Cl), aluminum (Al), chromium (Cr), iron (Fe), and oil content. It follows that any waste treatment technology must be capable of ensuring the liquid phase dissolved constituents do not come in contact with groundwater or soil. Solid phase chemical parameters of concern were: sodium (Na), potassium (K), magnesium (Mg), copper (Cu), zinc (Zn), molybdenum (Mo), Vanadium (V) and lithium (Li), titanium (Ti), barium (Ba), and strontium (Sr). All metals were, on average, higher in drilling wastes than in typical Alberta soils. These parameters are normally associated with drilling fluid additives. Oil-based mud wastes averaged 6.62% oil in the solid phase, while water-based mud wastes averaged 0.5%.

3. Mechanisms

3.1. Environmental Consequences of Drilling Fluid Disposal on Land

- The consequences of metals associated with drilling fluid additives for plant growth were studied in detail by Nelson, Liu and Sommers (1980), who conducted pot trials on mud-treated agricultural soils with Swiss chard (Beta vulgaris) and ryegrass (Lolium perenne L.). They found yields reduced by as much as 80% when high metals contents were present in water-based mud. Cadmium (Cd), Zn, Cu, lead (Pb), and arsenic (As) were incorporated in plant tissues in concentrations up to 44 times normal when these metals were present in the rooting zone, when compared to plants in unamended soils.

- Danielson, Okazawa and Ceroici (1989) conducted field studies of the effects of oily sludge on plant growth. They found that yields of Samson variety barley from soils treated with 3% to 5% oily sludge ranged from 53% to 83% of yields from untreated control plots. Plot to plot variability was much greater among treated crops than it was for the control plots. They also found that during the growing season, Samson barley incorporated very little hydrocarbon materials through their roots. This suggested that biodegradation of hydrocarbons occurs almost exclusively by soil organisms and very little, if any, uptake by plants occurs during this process.

3.2. Stabilization

- Conner et al (1992) provide a detailed discussion of the chemistry of stabilized wastes. They discuss the key components and the properties of each that pertain to leachability of metals, other inorganic chemicals and organic chemicals. They provide substantial detail on the influence of pH effects on solubility of metal hydroxides (the form most metals take within the cement matrix) and water/cation interaction in pore water.
  Conner and his colleagues compare metal behaviour within the cement matrix with their behaviour in contaminated soils and sludge. They also note that while some organic compounds are chemically altered and tightly fixed, some loss of VOC occurs during the mixing phase of the treatment process. Further work was recommended on this issue. They present two successful case histories, a lead-acid battery processing site and an oily/PCB waste pond reclamation project.
- Ivey et al (1992) confirmed, through extensive experimentation, that chromium III and chromium VI remain fixed in the cement matrix following leaching.
- The primary stabilization mechanism is chemisorption or adsorption. Haggerty et al (1994) have shown that chromate, selenate and sulfate can be effectively fixed in a common zeolite known as heulandite or clinoptilite modified with a surfactant. Li et al (1998) show that surfactant modified zeolite (SMZ) is also stable over the long term. Li et al (1997) also demonstrate that once adsorbed into the heulandite (clinoptilite) structure, can be defended against attacks by chloride, bisulfate and bromide counterions, all of which may be present in waste drilling fluids.
- Coal fly ash, a well known pozzolan, has been shown to be an effective adsorbent for mercury, copper, cadmium, and lead by Apak (2000) in his interim report to the NATO/CCMS pilot study on the evaluation of demonstrated and emerging technologies for the treatment and clean up of contaminated land and groundwater (web site www.nato.int/ccms/s13/report/interim00.htm). the study showed very high adsorption efficiencies (>99.9%) and that adsorption is essentially irreversible. Results for radionuclides were mixed. Concrete compressive and shear strengths were not significantly affected until 20% waste material was added, after which strength declines.
- Further support that strontium and cesium are preferentially adsorbed to an unspecified pozzolan is presented by Cetin and Mehta (1999). Experimentation over a range of environmental temperatures showed the adsorption reaction to be only slightly beneficially sensitive to increases in temperature.

3.3. Encapsulation

- Porter et al (1995) have conducted an extensive and intensive review of encapsulation technologies and found Portland cement and lime/pozzolans, sulphur polymer cement, phosphate ceramics and polyethylene to be effective for protection of the environment and human health for periods exceeding 100 years. Evidence from antiquity show pozzolan-treated concrete to be stable for in excess of 2000 years.
- Zhao et al(1999) have determined pore fluid chemistry to be a significant factor in controlling concrete permeability. The presence of sodium nitrate appears to ensure the permeability remains tight.
- Janotka and Stevula (1998) found treatment of Portland cement/bentonite mixes with zeolite both increased the strength of the product solid and improved resistance to chemical attack by sulphate. The product continued to be stable after 365 days of curing.

3.4. Treatability Testing

- Caldwell et al (1992) describe an approach to developing a specific stabilization protocol for inorganic residues with trace levels of organic contamination. They describe an example using metal finishing residues spiked with six organic compounds, two of which contain Volatile Organic Compounds (VOC). They used pulverized activated carbon as the adsorbent, drilling mud as the stabilizer and Portland cement as the solidification agent. An optimization procedure is described and performance working curves are constructed for the test substance.

4.

QA/QC for Stabilized/Encapsulated Drilling Waste

- Stegemann and Coté (1991) is the definitive work on assessment of suitable methods for the characterization of multiphase wastes and the evaluation of stabilized/solidified waste treatment products. The evaluation included the primary standard method for determining the leachability of a monolith (the USEPA Toxicity Characteristic Leaching Procedure (TCLP)). The report is based on an impressive review of the literature, which was both extensive and intensive, and on extensive laboratory experimentation conducted at the Wastewater Research Centre of Environment Canada, the Alberta Environmental Centre, the United States Army Corps of Engineers Waterways Experiment Station and Louisiana State University. The work included error analysis for each method and contains recommendations for quality control of measurements.
- A set of test methods were recommended for adoption by Governments for the assessment of solidified waste products and the evaluation of methods for particular wastes. The outcome from this work was the “Proposed Evaluation Protocol for Cement-based Solidified Wastes” (see Section 1). A copy of the TCLP is included under Section 4 in this compendium.
- Beckefeld (1992) describes a QA/QC program undertaken by Heitkamp Umwelttechnik GmbH, Bochum, Germany of solidification of a wide range of industrial wastes. Three standard methods are used in testing solidified wastes against a set of criteria mandated by the President of Munster. The battery of solidification product tests in the protocol are coefficient of disintegration; uniaxial compressive strength (DIN 18 136); and hydraulic conductivity (DIN 18 130). Long term monitoring is conducted for two years following completion of the project. It consists of phreatic pressure testing to monitor seepage into the stabilized waste body, remotely monitored neutron probes within the waste body and analysis of leachate recovered in situ (DIN 38414-S4). Leachate quality is compared to untreated soil leachate. Results have been very good, with polyaromatic hydrocarbon (PAH) levels in both leachate and surface water reported as very low.
- Samsonek et al (1998) concluded that leachability test results are sensitive to sample volume, from which they conclude that the surface area exposed to meteoric water in landfills may be a significant issue with respect to leaching of controlled substances.
- Sullivan et al (1998) have used Raman spectroscopy to confirm ion exchange adsorption of chromate to surfactant modified zeolite (SMZ) molecular structures.

5.

Case Histories of Drilling Waste and Oily Waste Stabilization/Encapsulation

- MacKay and Emery (1992) describe four cases where waste solidification/stabilization has been used successfully to fix metals and hydrocarbons. The first involves low level PCB contaminated road pavement (initial PCB concentration was 21.5 µg/g) at Clear Lake, Ontario. Following laboratory screening trials, a mix of 10% Portland cement (dry soil basis) was added to the contaminated material. An alternative mix consisting of 3% Portland cement and 12% cement kiln dust was also evaluated. Ontario Ministry of Environment criteria were used for the treatment product, which were met by both mixes. The final volume of the monofill was about 29% greater than the volume of the contaminated soil. Cost of treatment was $\$104.93/m^3$.

  The second case history was remediation of steel plant sludge in Hamilton. The sludge was an oily, high water content organic-metallic-lake bottom ooze with a bulk density of 1300-1650 kg/m^3 and loss on ignition of between 7% and 31%. The sludge had been further contaminated by municipal sewage. Following screening trials, an 8% to 12% mix of unslaked lime kiln dust plus 3% to 5% slag cement was added to the sludge and mixed vigorously using ready-mix trucks. The stabilized product passed the Ontario Ministry of Environment criteria for fill applications or landfilling. Cost was $8.00 per tonne using byproduct stabilizing agents.

  Case history three was a lead-acid battery reclaiming operation slag in Quebec. Following screening trials, a 10% mix of slag with lime-free Portland cement proved adequate to pass the Quebec leachate extraction test. No costs were supplied.

  The fourth case was a former fly ash fill site at an Ontario Hydro thermal generating plant. The fly ash was not considered to be an environmental hazard, however there was an aesthetics issue and it also had some economic value as a construction material. Following screening trials, a mix of dolomitic lime kiln dust was selected. The product had the desired handling and re-use properties and passed the Ontario leaching trials without problems.

- Collins and Luckevich (1992) describe three successful solidification projects: an acid mine spoil which was treated and re-used as backfill in the mine workings; waste glass as a concrete additive for construction; incorporation of automobile tire crumb into concrete and re-use as a construction material and flue gas desulphurization derived gypsum as an additive in the production of Portland cement.

- Akinlade et al (1997) is a case study published on the Australian EIA Network website (www.environment.gov.au/epg/eianet/case_studies/cs_87.htm) concerning drilling waste disposal in Nigeria using Portland cement and a proprietary additive, Geosta-A. Following screening trials, a 1:12 (8%) mix of Portland cement and 0.1 to 0.3% Geosta-A were applied to the cuttings, which met Department of Petroleum Resources and Federal Environmental Protection Agency guidelines, except for chloride.

- Boyce and Alskog (undated) describe a series of mercury treatment options for contaminated soil at the Brookhaven National Laboratory, New York. It may be found at the State University of New York, Sunnybrook website (www.pbisotopes.ess.synysb.edu/lig/Conferences/Abstracts99/boyce.htm). They found stabilization to be cost efficient ($40 to $2000US per ton) and readily available. Processing rates are also high (40 tons per day) and removal efficiencies were also very high (from 260 mg/kg in the untreated contaminated soil to TCLP Hg values of less than 0.2 mg/l in leachate.

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