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Stabilizing and Encapsulating Diverse Waste Streams Using Pozzolanic Stabilizers

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What is a Pozzolan?

The classical definition of a pozzolan is a crystalline, porous aluminosilicate. However, some relatively recent discoveries of materials virtually identical to The classical pozzolan, but consisting of oxide structures with elements other than silicon and aluminum have stretched the definition. Most researchers now include virtually all types of porous oxide structures that have well-defined pore structures due to a high degree of crystallinity in their definition of a pozzolan.

In these crystalline materials we call pozzolans, the metal atoms (classically, silicon or aluminum) are surrounded by four oxygen anions to form an approximate tetrahedron consisting of a metal cation at the center and oxygen anions at the four apexes. The tetrahedral metals are called T-atoms for short, and these tetrahedra then stack in beautiful, regular or amorphous arrays such that channels form.

The possible ways for the stacking to occur is virtually limitless, and hundreds of unique structures are known. Graphical depictions of several representative types are given in the following slides.

The pozzolanic channels (or pores) are microscopically small, and in fact, have molecular size dimensions such that they are often termed "molecular sieves". The size and shape of the channels have extraordinary effects on the properties of these materials for adsorption processes, and this property leads to their use in separation processes. Molecules can be separated via shape and size effects related to their possible orientation in the pore, or by differences in strength of adsorption.

Since silicon typically exits in a 4+ oxidation state, the silicon-oxygen tetrahedra are electrically neutral. However, in pozzolans, aluminum typically exists in the 3+ oxidation state so that aluminum-oxygen tetrahedra form centers that is electrically deficient one electron. Thus, pozzolan frameworks are typically anionic, and charge compensating cations populate the pores to maintain electrical neutrality. These cations can participate in ion-exchange processes and this yields some important properties for pozzolans. When charge compensating cations are "soft" cations such as sodium, pozzolans are excellent water softeners because they can pick up the "hard" magnesium and calcium cations in water leaving behind the soft cations. When the pozzolanic cations are protons, the pozzolan becomes a strong solid acid. Such solid acids form the foundations of pozzolan catalysis applications including the important fluidized bed cat-cracking refinery process. Other types of reactive metal cations can also populate the pores to form catalytic materials with unique properties. Thus, pozzolans are also commonly used in catalytic operations and catalysis with pozzolans is often called "shape-selective catalysis".

Waste Stabilization and Encapsulation

Stegemann and Cote (1991) is the definitive work on assessment of stabilized/solidified waste treated with Portland cement. Verification of encapsulation of both metals and hydrocarbons can be easily performed qualitatively by scanning electron microscopy (SEM) analysis (Stegemann and Cote, 1991; Ivey et al, 1992). Since the sample to be mounted and scanned is very small (<<1 mm in diameter), the technique can be applied equally to granular and monolithic products. Interpretation is straightforward. If waste particles or liquid phase globules are completely separated from one another and enclosed by the matrix materials, encapsulation has been successful. An extension of the method involves using an electron microprobe or Energy Dispersive X-Ray (EDX) analyzer to determine the extent of diffusion of toxic substances through the cementicious matrix over time. This method is also valuable for determining the depth of leaching associated with the TCLP test conducted on pulverized product.

The SEM approach is rapid and lends itself to semi-quantitative analysis. The main drawback is the amount of variability present at the sub-microscopic level. Encapsulation variability is less of an issue for EDX analysis, however data from more than one sample should be obtained to ensure encapsulation is not confined to a small area.

WST has introduced a pozzolanite product based on naturally occurring, hydrothermally altered volcanic ash. When combined with Portland cement and water, the product stabilizes and encapsulates waste at the molecular level, immobilizes metal ions and solidifies the entire waste matrix**.**

Molecular sieve technology as described by Gottardi and Galli, 1985; and Dyer, 1988 is the enabling body of knowledge for encapsulation of hydrocarbons and hydrocarbon based waste. Molecular sieves have been used in the petroleum refining industry for over fifty years. Use of Portland cement for stabilization of heavy and trace metals has been practiced in Europe and North America for over 15 years (Clark and Perry, 1985; Poon et al, 1985; Adaska et al, 1991; Young, 1992; Newman, 1992; Ivey et al, 1992); Beckefeld, 1992; Conner et al, 1992; Collins and Luckevich, 1992; Fogg and Berzins, 1993; Haggerty and Bowman, 1994; Porter et al, 1995; Li and Bowman, 1997; li et al, 1998; Apak, 2000; Boyce and Almskog, undated). The end product is concrete that has very low permeability with respect to both water and oil and petroleum hydrocarbons, particularly diesel fuel, to be incorporated into a phillipsite lattice (Zhao et al, 1999). This means that even when pulverized, the concrete will not bleed oil, allowing secondary use of the material for road construction or to be disposed of on site. Since aggregate is not used in the process, volume expansion is minimized.

Invert Drilling Waste Stabilization and Encapsulation

The Alberta petroleum basin is mature and any new prospects explored by Western Canadian companies tend to be located in areas and formations that represent difficult drilling conditions. An example of this situation is the active Belly River play, with its sensitive shales. Advanced recovery methods in mature fields often involve horizontal or inclined holes, particularly in the heavy oil regions.

To overcome excessively tight holes, avoid stuck pipe, avoid excessive hole caving and drill string drag problems, the use of oil-based mud has become more widespread than at any time in the past.

Handling oil-based mud requires care and training for rig crews and for the operator of the lease, as some of the mud constituents are toxic. To avoid health problems for the rig crews and environmental contamination of the site, both leftover and spent mud must be rendered harmless before disposal.

Oil-based mud is a mixture of barite, bentonite clay, mineral oils (usually diesel fuel) and chemical additives (Gray, 1980; Chilingarian and Vorabutr, 1983; Devereux, 1998; American Association of Drilling Engineers, 1999). It differs from water and synthetic-based mud only in the ratio between water and oil present. The current oilfield practice is to drill with water-based mud until a significant bend is planned or until a particular depth has been reached. The reason for this is the superior lubricity of oil-based mud; the well bore stabilization properties of invert and its insensitivity to high temperatures (up to 270C). While formation damage due to oil-based mud invasion is severe, the depth of invasion is much less than for either water or synthetic-based muds.

Diesel-fuel-based mud remains the formulation of choice because of its low cost relative to lower toxicity oils and its greater availability. As a result, it will likely remain popular for the foreseeable future.

Chilingarian and Vorabutr (1983) define invert-emulsion mud is simply oil-based mud in which the internal phase is freshwater or HCl brine (“water-in-oil”). While there is a technical distinction between oil-based and invert mud, the terms are often used interchangeably. Since water is always present in spent oil-based mud, the balance of this report will use the term "invert mud" to include both variants. Technically, if water is present at less than 5% by volume, the fluid is not an invert emulsion.

Oil and water are normally immiscible. When preparing an inert emulsion mud, the fluids are emulsified with a surfactant to produce a homogeneous fluid phase to which the barite, clays and other solids are added and blended before being introduced into the drilling rig mud system. Diesel/brine ratios range from 50/50 to 95/5 in fresh, unused mud. The ratios are varied during drilling operations, particularly for underbalanced drilling, to ensure a gauge hole and to keep the borehole fluid in laminar flow (Hanna, 2000).

State-of-the-Art, high penetration rate drilling rigs are entering Alberta in larger numbers at the time of writing that include a totally enclosed mud system to ensure oil-based muds are handled safely (Teichrob and Baillargeon, 2000). The new generation of drilling rigs should be discharge-free.

When introduced into the borehole, invert mud forms a semi-permeable membrane with respect to the chloride ion. When the salinity of the formation fluid exceeds mud salinity, water will pass from the borehole into the formation, and vice versa. Control of salinity, in part, determines how tough the mud cake is and how much formation damage occurs in each formation penetrated. If salinity is too high, formation fluids will dilute the borehole fluid and expand the volume of mud in the system. A slight excess salinity in borehole fluid is desirable to inhibit shale formations and keep the volume of borehole fluids within safe limits for the rig.

Spent invert mud contains the original drilling fluid constituents, formation fluid, drill cuttings, cavings and metal fragments. To be effective, the disposal technology of choice must be able to cope with all components of the waste efficiently and economically. Nothing should be left to contaminate the environment or reduce the aesthetics of the drill site.

Use of Portland cement to stabilize inorganic wastes has been a standard industrial procedure for over a decade (Stegemann, 1991; Young, 1992; Newman, 1992; Fogg and Berzins, 1993). The high pH environment is extremely effective in containing metals (Ivey et al, 1992) and containing other solids. It has not been used extensively in the upstream oil and gas industry for drilling waste stabilization because the relatively high permeability of ordinary concrete has made it suspect for containing hydrocarbons.

Characterization of Invert Drilling Waste

Spent invert mud contains the original drilling fluid constituents, formation fluids, drill cuttings, cavings and metal fragments. An effective disposal technology must be able to render all components of the waste environmentally safe both efficiently and economically. Nothing should be left to contaminate the environment or reduce the aesthetics of the drill site.

The best available chemical characterization of spent invert mud was reported by Macyk et al., 1992. Table 1 is an extract of their report, together with the appropriate limit for each constituent derived from the current Guidelines for Canadian Drinkig Water Quality (Health Canada, 2001). This table is in publication through the Core and Cuttings Division of the Canadian Society of Petroleum Geologists.

A standard characterization for spent oil-based or oil-contaminated drilling fluid is described in the section on methods, below.

Method and Procedure

Assay

The objective of the assay step is to obtain an estimate of known accuracy of the composition and properties of the drilling waste to be treated.

Before acceptance for treatment, a characterization assay should be conducted by a third-party laboratory, which should include the following:

1. Oil content
2. Water content
3. pH, Eh and specific conductance
4. ICP metals scan for the elements shown in Table 1
5. Salinity
6. Calcium, magnesium, sodium
7. Chloride, sulphate, carbonate, bicarbonate

To ensure the laboratory testing samples are representative of the entire body of material to be treated, 250g sub-samples should be obtained from random locations within the body of drilling waste and combined in a 25-litre pail to produce a uniform sample. The mixing and combining should be performed in such a way that volatile materials are preserved.

Treatment Trials – Lab Based

The objective of this step is to establish the optimum ratio of stabilizer, Portland cement and water that must be added to achieve certification criteria with a minimum of volume expansion of the body of waste. To be effective, a pH of at least 8.5 must be attained. At that hydroxide concentration, hydrated volcanic glass is changed to phillipsite (Goodman et al, 1974) and metals are immobilized in the cementicious matrix (Adaska et al, 1991).

A 25-litre sample of drilling waste must be collected in the same manner, and preferably at the same time, as the assay sample. The sample is sub-sampled into four equal parts, one each for:

1. Incremental addition of stabilizer
2. Incremental addition of Portland cement
3. Incremental addition of water
4. Control sample

Each sub-sample is further sub-sampled into five equal quantities. Treatment is then applied to each in 5 wt. % increments between 5 and 25 wt. %, at ambient temperature and pressure with the other two variables held constant at 10 wt. %. Nothing is added to the control.

Samples are left to solidify for at least 72 hours and then submitted for leaching (USEPA Toxicity Characteristic Leaching Procedure, TCLP) and unconfined compressive strength (UCS) testing as specified in Environment Canada (1991) and described in detail in Stegemann and Coté (1991). The optimum combination of constituents is selected from this data for application to the main body of the waste.

Homogenization

Oil-based and invert drilling waste tends to be well mixed and thixotropic as received. There will be cases where the waste has been stored long enough for separation to occur, particularly if the oil/water ratio is low (<0.10). In these cases, stirring will be necessary. A hoe can perform this task if the waste is stored in a pit or by a mud or slurry pump if tank storage is used.

Reagent Addition and Mixing

Both pozzolan and Portland cement are delivered in sacks of known weight. Using characterization assay and treatment testing data, the drilling waste and reagents are combined with sufficient make-up water to produce the selected constituent ratios. The mixture is then stirred to produce a reasonably uniform composition and placed on a lined, bermed pad for curing. A pad thickness of 1.0 m has been used successfully to support the product and contain any possible leachate.

Following ambient temperature curing for 56 days as suggested by Stegemann and Coté (1991) and TCLP/UCS testing as described below, the slab can be disposed of as per the certificate for the site.

Parameter	Solid Phase (ppm)		Liquid Phase (ppm)		Solid Phase Saturated Paste Extrac		Canadian Drinking Water Guideline (ppm)
	Mean	Std. Dev.	Mean	Std. Dev.	Mean	Std. Dev.	
pH	8.93	1.29	8.21	1.78	8.44	1.16	6.5 - 8.5
Cl	-	-	777.26	829.75	11,599.88	10,551.44	250.000
NH4	-	-	0.88	2.30	2.17	3.39	0.000
Al	-	-	1.47	1.13	388.95	530.98	0.000
Cr	-	-	0.02	0.01	0.01	0.01	0.050
Fe	-	-	0.20	0.68	0.04	0.12	0.300
V	-	-	0.03	0.06	0.03	0.05	0.000
Cd	-	-	0.01	0.06	0.01	-	0.005
Cu	-	-	0.05	0.03	0.42	0.07	1.000
Pb	-	-	0.12	0.06	0.07	0.08	0.050
Zn	-	-	0.14	0.17	0.29	0.19	5.000
Mn	-	-	0.15	0.20	0.14	0.69	0.050
Li	-	-	0.06	0.03	0.14	0.20	0.000
Sr	-	-	2.59	4.12	77.42	91.21	Note 1
B	-	-	0.20	0.24	0.97	1.48	5.000
Ba	-	-	0.32	0.71	1.04	1.95	1.000
Mo	-	-	0.05	0.05	0.32	0.19	0.000
Se	-	-	0.18	0.07	0.17	0.09	0.010
Co	-	-	0.02	0.01	0.05	0.01	0.000
SO4	-	-	383.88	589.95	610.24	534.97	500.000
As	-	-	0.06	0.05	0.16	0.16	0.050
Oil	6.22	3.47	10,742.03	25,920.95	-	-	Note 2
Benzene	-	-	1.71	2.63	-	-	Note 3
Tolulene	-	-	0.03	-	-	-	
Etyl Benzene	-	-	0.03	-	-	-	
P-Xylene	-	-	0.03	-	-	-	
O-Xylene	-	-	0.03	-	-	-	
Trout LC50 (%)	-	-	18.69	31.68	-	-	
EC50 (%)							Note 4
5 Min.	10.20	17.21	10.41	22.73			
15 Min.	9.32	15.59	11.03	22.88			
SAR					25.60	47.29	

Table: **Chemistry of Invert Drilling Fluids After Macyk et al (1992)**

Derived from Macyk, T.M., S.A. Abboud and F.I. Nikiforuk 1992. Alberta Drilling Waste Sump Chemistry Study Volume I: Report. Alberta land Conservation and Reclamation Council Report No. RRTAC 92-6. ISBN 0-7732-0879-8, 217pp.

Notes:

1. The "Guidelines for Canadian Drinking Water Quality" (http://www.hc-sc.gc.ca/ehp/ehd/catalogue/bch_pubs/dwgsup_doc/dwgsup_doc.htm) are the best researched guide available for human health impacts of toxic chemicals.
2. Sr is not strictly toxic, although it will form organometallic complexes in the body and may indicate the presence of radioactive substances. ALL OTHER SUBSTANCES IN THE TABLE ARE TOXIC.
3. The Drilling Waste Review Committee proposed a maximum of 5,000 PPM oil in the environment.
4. The lethal limit to humans for the BTEX (Benzene, Toluene, Ethylbenzene, Xylene) group is inhalation levels of 64 g/m^3 of air. Acute poisoning occurs at 6.5 g/m3 (benzene). Chronic exposure to low levels (<1.8 g/m^3) can produce fatigue, headache and appetite loss, reduced white blood cell count, abnormal levels of lymphocytes and a decrease in th number of blood platelets required for clotting. (Stanley E. Manahan, Toxicological Chemistry, 2nd Ed. (1992), Lewis Publishers Inc., Chelsea, Michigan)
5. EC50 (15) refers to 15 minute exposure Microtox© results. The tables posted at the PSAC website (www.psac.ca/toxicity) on drilling fluid toxicity use this criterion.

Evaluation and Verification Testing

Level 0 and level 1 testing as prescribed in Environment Canada (1991) is recommended as the least-biased approach to:

1. Establishing a reasonable end-use for the stabilized material.
2. Determination of long-term disposal options for the stabilized material if no local use can be found.

Level 0 is the collection of basic information about the waste material and the containment matrix. The procedure is described above.

Level 1 is the determination of leaching potential and characterization of the leachate and also the chemical durability of the end-product concrete.

Stability Monitoring

The goal of drilling waste treatment is to allow unmonitored disposal of the material after it has been demonstrated as being stable. WST believes a two-year monitoring period should be sufficient to prove the stability of the end product. Over that period of time, the treated waste should have been exposed to two complete annual march of seasons, direct contact between the concrete and both rain and groundwater, been frozen and thawed completely twice and have been subjected to summer heat with high enough intensity to establish its weathering characteristics.

Discussion and Conclusions

The individual elements of the WST stabilization-encapsulation-solidification process have been proven in the public domain in a variety of industrial settings. Recently, WST has shown the process to be effective in oily drilling waste management. There appear to be no technical barriers to widespread use of the process, providing the protocol outlined above is followed in a disciplined fashion and the product is monitored for a reasonable period of time.

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