

## ORGANIC WASTE TREATMENT WITH ORGANICALLY MODIFIED CLAYS

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## ABSTRACT

A relatively new technology for the retention and adsorption of organic pollutants involves the use of organically modified clays. The accessible surfaces within the crystalline structure of clay minerals are chemically modified with organic derivatives such as alkylammonium ions. This clay modification imparts an organophilic character to the clay. The clay surface is thus rendered suitable to adsorb organic molecules.

Clays such as bentonite have been used for many years as pond and landfill liners because of their low permeability to water. The low permeability of these clays has been shown to be affected adversely by fluids containing organics. Organically modified clays allow an extension of clay barrier technology into organic systems. As a result of the affinity between organically modified clays and organic pollutants, applications for their use in waste treatment and remediation have evolved. These applications include:

- 1) Waste stabilization - organically modified clay is mixed with organic wastes and then cementing agents to produce a solidified matrix, resulting in reduced leachability of the organics from the stabilized matrix.

- 2) Water treatment - organically modified clay is used for treatment of ground and surface water to remove organic constituents within the waste stream.

- 3) Spill control - organically modified clay can be distributed on water or soil surfaces to sorb organic liquids as necessary for spill control.

- 4) Tank farm liners - organically modified clay can be used in liner systems for fuel oil storage tanks.

- 5) Hazardous waste liner systems - organically modified clay can be used as a barrier layer component within liner systems for hazardous waste storage and disposal sites.

TABLE 5, SUMMARY OF RESULTS OBTAINED WITH THE 1 TON/HOUR  
EXTRA SOL UNIT - PCP REMOVAL

TYPE OF WASTE	TYPE OF SOLVENT	Initial PCP Concent. (ppm)	Final PCP Concent. (ppm)	% Removal (%)
porous gravel	# 2	8.2	<0.82	>90
porous gravel	# 2	81.4	<0.21	>99.7
porous stones	# 2	38.5	19.5	50
activated carbon	# 2	744	83	89

#### Disclaimer

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## INTRODUCTION

The use of organically modified clays in hazardous waste management applications offers a significant new and untapped potential. These clays may be used in the stabilization of organic wastes and organically contaminated soils, for waste water treatment, for oil spill control, for liner systems beneath fuel oil storage tanks, and as a component within liner systems of hazardous waste storage treatment and disposal facilities. Organically modified clays (organophilic clays) may be employed in each of these systems to adsorb organic waste constituents, enhancing the performance of these applications. This paper first describes the nature of organophilic clays, and then discusses their application in each of the five areas.

## ORGANICALLY MODIFIED CLAYS

The production of organically modified clays begins with the use of a natural clay mineral. The clay minerals most commonly used in this process are smectites (montmorillonite and hectorite) and attapulgite (palygorskite). Since the structure of each of the base clays differs, the performance of the modified clays will likewise vary. Detailed descriptions of these clays is found elsewhere (6, 11)

### Organic Modification of Clays

The investigation of clay-organic interactions began over 50 years ago. An early study reacted organic bases and their

salts with montmorillonitic clays and presented evidence that an ion exchange reaction had occurred (6). Similar early experiments using organic chemicals in montmorillonitic clay demonstrated that the exchangeable inorganic cations could be replaced by organic cations, and that uncharged polar compounds could enter the inner layer region without the release of cations (13). It was also found that bentonite, after reaction with certain organic compounds, gains the properties of swelling and dispersing in organic fluids (8). These studies describe the clay-organic interactions which impart the organophilic characteristics upon the modified clay. Since that time, these interactions have proven to be effective and, in many cases, commercially viable in transforming a naturally hydrophilic clay into an organophilic clay.

A number of chemical interactions between the clay and organic compound were identified in the organic modification of clays. The primary reactions which occur are adsorption, intercalation, and cation exchange. Additional reactions include ion exchange, anion exchange, protonation of organic molecules at the clay surface, hemisalt formation, ion-dipole coordination, hydrogen bonding, pi-bonding, entropy effects, and covalent bonding (10). It is beyond the scope of this paper to discuss in detail these reactions, which influence the organic modification of clays.

To produce an organically modified clay, an unmodified clay mineral is reacted with an organic compound. In this

process, a cationic surfactant, such as quaternary ammonium, replaces the exchangeable inorganic sodium, calcium and/or magnesium ions on the negatively charged surface of the clay. In this reaction, the clay's nature is converted from a hydrophilic to an organophilic condition. Reaction of the clay with the appropriate organic cation will result in a modified clay which will swell and disperse in the presence of a variety of organic liquids.

The organic compounds most commonly used to modify clays are quaternary ammonium salts. A quaternary ammonium salt is a form of an organic nitrogen compound in which the molecular structure includes a central nitrogen atom joined to four organic groups along with an acid radical. They are all considered cationic, surface active coordination compounds and tend to be adsorbed on surfaces, thus the term surfactants. The most commonly employed types of quaternary ammonium compounds used to modify clays are dimethyl ammonium, methyl benzyl ammonium, dibenzyl methyl ammonium, and benzyl dimethyl ammonium quats.

#### Manufacture of Organically Modified Clays

Organically modified clays are manufactured using either a dry process or a wet process. In a wet process, the unmodified clay is mixed with water, forming a slurry. The resulting slurry is centrifuged to remove inert, non-clay minerals. The supernatant, which contains ultra-pure clay, is then reacted with the specified organic compound. The mixture is filtered, dried and ground.

In the dry process, limited amounts of water are first added to the unmodified clay. The clay is then reacted with the organic compound in a mixer, pug mill or extrusion device. Finally, the reacted material is dried and ground. Since centrifugation is not performed in the dry process, some impurities still exist in the finished clay product. Additional detail regarding the clay manufacture can be found elsewhere (1, 5, 12).

#### Adsorption by Organophilic Clays

Organically modified clays are suitable media for the adsorption of soluble organic compounds from dilute aqueous solutions. This adsorption occurs through electrostatic/hydrogen bonding forces at the hydrophilic sites, and by van der Waals forces at the organophilic sites of the organophilic clay. The following factors affect the adsorption of organic compounds from dilute aqueous solutions by organophilic clays: a) the nature of the adsorption sites, b) the nature of the organic molecules to be adsorbed, c) spatial considerations, d) thermodynamic quantities, and e) solubility of the adsorbate in the solvent (4). Portions of the organically modified clay surface which were not modified during the organic modification process are still hydrophilic. As a result, adsorption by electrostatic/hydrogen bonding with the hydrophilic portion of the adsorbate molecule occurs at these hydrophilic clay sites. On the remainder of the clay surface, which is organophilic, van der Waals bonding occurs. Since adsorption occurs at both

types of sites on the clay surface, a balance between the organophilic and hydrophilic clay sites optimizes the adsorption capacity of the clay (4).

#### LABORATORY TESTING PROGRAM

The adsorption capacity of a number of commercially available organically modified clays was quantified utilizing the free swell, or sedimentation, test. In each of these tests, 50 milliliters (ml) of the test fluid were poured into a 100-ml graduated cylinder and 2.5 grams of the clay were then rained into the fluid. The clay typically settled to the bottom of the graduated cylinder. The free swell volume was recorded after 24 hours. The laboratory test procedure for this free swell test was modified from laboratory procedures previously developed (7). The major modification from the original procedure was the reduction in the quantity of clay and test fluid utilized. These investigations parallel a study published earlier in which a single organically modified clay type was evaluated (8, 9). This swell volume is only an indicator of the clay's adsorption capacity, and may not adequately quantify the performance of the clay in the applications discussed herein.

The clays and their manufacturers are listed in Table 1 along with pertinent information regarding the manufacture of the organophilic clays. The organic compounds reacted with the base clays are quaternary ammonium salts, primarily dimethyl di(hydrogenated tallow) ammonium chlorides. Tallow is an animal fat with each

organic molecule containing 16 to 18 carbon atoms.

Ten test fluids were used in these studies: acetic acid, acetone, aniline, carbon tetrachloride, deionized water, diesel fuel, hexane, kerosene, unleaded gasoline, and xylene. All of the fluids employed in the study were added in a concentrated form with each of the clays studied. The results of the free swell tests conducted for these studies, along with the density of each clay, are summarized in Table 2.

#### DISCUSSION OF LABORATORY TEST RESULTS

Examination of the average free swell volume enables a comparison between the various organically modified clays for a wide range of organic fluids. Viewing the data in this way, it is revealed that all of the organically modified clays have an average free swell volume between 11 and 35 ml. The wet process clays with the highest average free swell volumes are Baragel 3000 (34.3 ml), Benathix 1-4-1 (32.1 ml) and Tixogel SP (31.6 ml). The dry process clays with the highest average free swell volumes are PC-1 (23.8 ml) and TS-55 (23.1 ml). The two unmodified clays, bentonite and attapulgite, have the lowest average free swell volumes, indicating they did not swell appreciably in the presence of the concentrated organic test fluids.

In summary, it is believed that these tests provide a useful way to rapidly and quantitatively compare the expected performance of organically modified clays. Note that clay cost must be evaluated in comparison to performance in

selecting a particular clay for a particular application.

## APPLICATIONS

With the ability of the organically modified clays to adsorb organics, a number of applications for organically modified clays in pollution control and hazardous waste site remediation have been identified. These applications range from those fully developed and in the marketplace to those recently proposed. The following section discusses several applications for organically modified clays.

### Waste Stabilization

The remediation of organically contaminated soils and wastes employing stabilization and solidification techniques has become increasingly widespread. The stabilization process is designed to maximize shear strength and minimize the rate of leaching of hazardous constituents from the stabilized matrix into the environment. Conventional stabilization techniques, such as cement and flyash, are usually limited to inorganic, metal-bearing wastes. For organic wastes, modified clays have been employed to adsorb organic constituents. Preliminary laboratory data indicate that organophilic clays are effective as stabilization agents (12).

When used in conjunction with conventional cement-based or pozzolanic additives, organically modified clays are effective in reducing the mobility of organic constituents from the stabilized matrix. Reductions in the

mobility of organics has been demonstrated and is the subject of several patents (2, 3). Organically modified clays are first mixed with the waste to adsorb the organic constituents. In this manner, the organics are chemically bound within the organoclay, thereby reducing the organic interference with the normal cement reactions and lattice formation. The clay, with organic contaminants bound within the clay structure, is then macroencapsulated in a cementitious matrix formed by a cement or pozzolan. This technique, which utilizes an organophilic clay in conjunction with a cement product, is employed by the Silicate Technology Corporation of Scottsdale, Arizona for the stabilization of organic hazardous wastes. In this technique, the leaching potential of the organic constituents is decreased through the use of organophilic clays as compared to techniques using only cement or pozzolan in the stabilization process.

### Water Treatment

Organically modified clays are used to treat organically contaminated waste water. In this process, the aqueous solution is filtered through organically modified clay and the organic contaminants are adsorbed by the clay. Unlike activated carbon, which adsorbs organic contaminants through surface related phenomena, organically modified clays swell as the organic contaminants are sorbed into the clay structure. Thus, the organic molecules of the contaminant preferably partition into the organic phase of the organoclay instead of the aqueous phase

(2). Volatile organics are poorly adsorbed, whereas oils and greases are readily adsorbed as a result of their differing partition coefficients.

Organically modified clays are typically used along with other water treatment technologies. For example, when used in a treatment system upstream of activated carbon, the carbon life is greatly extended as a result of the removal of high molecular weight organics. Two commercially available products, Calgon's Klenisorb 100TM and Electrum's Organosorb, contain an organically modified clay to remove organics from a waste stream. The organophilic clay in these products is used with an anthracite filter media to provide an effective column filtration medium in water and waste water treatment. The combination of this mixed media and granular activated carbon adsorption facilitates the removal of a broad range of both soluble and insoluble compounds. Shown on Figure 1, are the influent and effluent oil concentrations for an oily steam condensate filtered with a mixture of Organosorb and anthracite filter media. The treatment effectiveness is demonstrated on Figure 1 and an annual savings of about \$150,000 per year was projected for a 500 to 600 gpm system (14). In another application, the organically modified clay removes non-volatiles prior to air stripping, thereby increasing the efficiency of the system.

### Spill Control

The organophilic nature of organically modified clays make

them suited for use in spill control applications. As demonstrated in the laboratory studies, these clays will either float on or sink to the bottom of an aqueous solution, depending on the nature of the organic modification. For example, an oil spill on water can be sorbed by the organoclay and held within the clay crystalline structure for cleanup and disposal. For a spill where the materials sink, the clays could likewise be selected to sink through the water and sorb the spilled fluid.

### Tank Liners

Fuel oil storage tanks are typically surrounded with a liner and berm system to contain the fuel oil should a leak occur. As a result of the impervious nature of the liners used in these systems, they also contain precipitation. As an alternative to conventional liner systems, it is proposed that organically modified clays be used. An organically modified clay liner would permit precipitation to flow downward through the liner without accumulating in the containment system. In the event of a tank leak, the clay will swell in the presence of the fuel and form an impervious barrier layer. This would prevent migration of contaminants into the subsurface. It may also be possible to use organically modified clays as secondary containment barriers beneath/around underground storage tanks in a similar manner.

### Landfill Liners

The technology for landfill liners has been evolving in recent years to include

multimedia barrier layers to optimize the environmental protection. Presently, liner systems include both geomembrane barrier layers and natural clay barriers. The greatest environmental concern at present is that both geomembranes and natural clays may be subject to degradation in the presence of organic contaminants. Further, organic contaminants may migrate through these materials in response to chemical diffusion gradients. It is proposed that the liner system include a barrier layer composed of an organically modified clay. In this way, the multimedia liner system would have superior performance in the presence of organic contaminants. The sorptive capacity of these materials would significantly reduce the rate of organic contaminant transport across the liner system.

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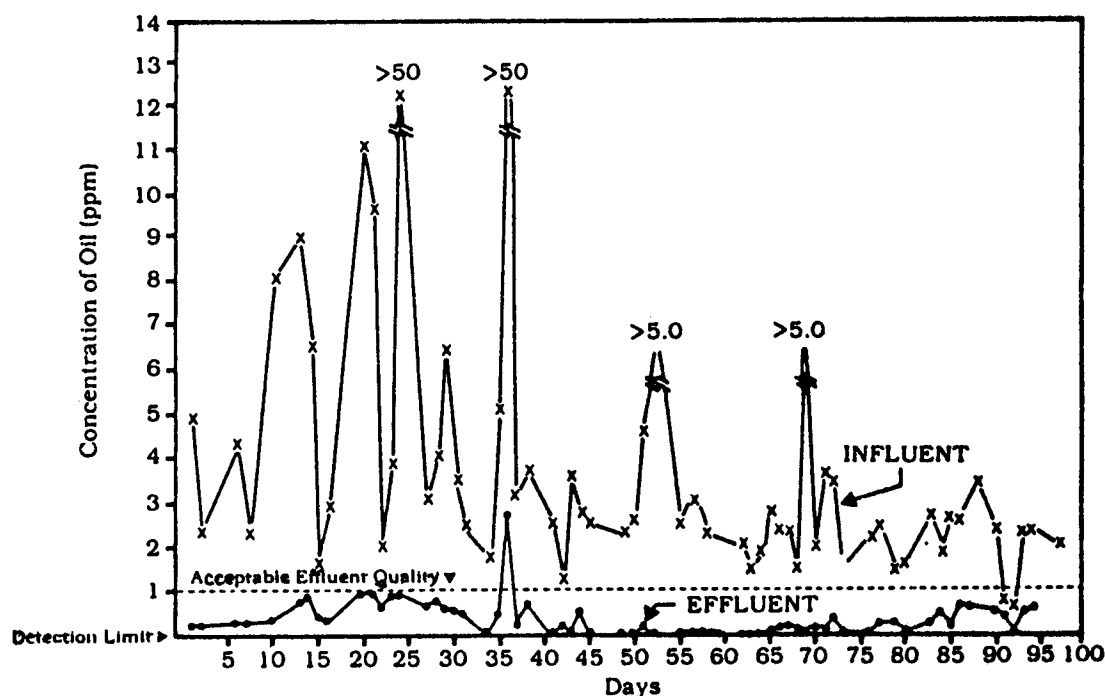


Figure 1. Influent and Effluent Concentrations (from Electrum, Inc.)

Table 1. Summary of Clay Information

						CARBON TETRA-						
	BULK	DEIONIZED	ACETIC							DIESEL	UNLEADED	
CLAY	DENSITY	WATER	ACID	ACETONE	ANILINE	CHLORIDE	HEXANE	KEROSENE	XYLENE	FUEL	GASOLINE	AVERAGE
	(g/cm <sup>3</sup> )	(ml)	(ml)	(ml)	(ml)	(ml)	(ml)	(ml)	(ml)	(ml)	(ml)	(ml)
Attapulgate	0.52	15.5	7.0	6.5	8.0	6.0	7.0	7.0	7.0	7.0	6.0	6.8
Bentonite	0.78	34.0	4.5	6.0	4.5	5.0	5.0	5.0	5.0	4.0	4.0	4.8
Attatone J	0.25	16.0	8.0	19.0	14.5	23.0	15.0	17.5	26.0	25.0	20.0	18.7
Attatone T	0.58	8.5	13.0	20.0	7.0	11.0	8.0	8.5	12.0	11.0	11.5	11.3
BC 90	0.40	8.5	11.0	11.5	7.5	7.0	18.0	18.0	20.0	20.0	29.0	15.8
Bondtone	0.53	7.0	8.0	13.0	7.5	0.0	40.0	20.0	36.0	19.0	21.0	18.3
Claytone APA	0.24	14.0	19.0	17.5	23.0	0.0	9.0	21.0	50.0	36.0	36.0	23.5
Claytone 40	0.47	0.0	13.0	14.0	12.0	0.0	12.0	22.0	48.0	46.0	29.5	21.8
ZHT	0.52	6.0	8.0	11.0	8.0	15.0	47.0	27.0	43.0	20.0	18.0	21.9
ZHT Aberdeen	0.51	10.0	10.0	16.0	8.5	3.0	12.0	18.0	18.0	20.0	27.0	14.7
P-1	0.36	9.0	12.0	13.5	11.0	0.0	13.0	18.0	27.0	21.0	22.5	15.3
P-11	0.34	8.0	9.0	10.0	9.0	25.0	25.0	22.0	25.0	18.0	20.0	18.1
P-40	0.51	7.0	9.0	13.0	8.0	33.0	13.0	26.0	20.0	16.0	31.0	18.8
PC-1	0.39	8.0	8.0	26.0	8.0	20.0	45.0	33.0	24.0	25.0	25.0	23.8
PT-1	0.44	7.0	9.0	22.0	8.0	10.0	15.0	32.0	22.0	28.0	29.0	19.4
Suspentone	0.50	10.0	11.0	15.0	9.0	6.0	28.0	35.0	33.0	17.0	28.0	20.2
TS-55	0.41	6.0	7.0	8.0	7.0	40.0	34.0	24.0	28.0	21.0	39.0	23.1
Tixogel DS	0.42	7.0	12.0	13.5	12.0	30.0	26.0	25.0	51.0	25.0	25.0	24.4
Tixogel EZ-100	0.49	6.0	11.0	12.0	11.0	20.0	47.0	30.0	20.0	20.0	15.0	20.7
Tixogel GB	0.53	7.0	9.0	11.0	8.5	0.0	13.5	24.0	20.0	20.0	13.0	13.2
Tixogel PE	0.44	7.0	11.0	20.0	16.0	0.0	10.0	15.0	25.0	20.0	35.0	16.9
Tixogel SP	0.43	11.0	14.0	15.0	13.5	25.0	18.0	49.0	50.0	50.0	50.0	31.6
Tixogel TE	0.42	8.0	13.0	16.0	13.0	0.0	16.0	37.0	51.0	50.0	50.0	27.3
Tixogel VP	0.50	10.0	12.0	15.0	12.0	28.0	10.0	19.0	20.0	49.0	35.0	22.2
Tixogel VZ	0.28	10.0	12.0	32.0	19.0	13.0	9.0	11.0	25.0	21.0	13.0	17.2
Bentone 27	0.55	8.0	12.0	14.5	16.0	7.0	5.0	6.0	22.0	12.0	8.0	11.4
Bentone 34	0.55	13.0	15.0	14.0	12.0	0.0	10.0	28.0	22.0	48.0	34.0	20.3
Bentone 38	0.46	15.0	18.0	15.0	14.0	43.0	9.0	18.0	39.0	32.0	10.0	22.0
Baragel 3000	0.43	16.0	16.0	20.0	14.0	30.0	42.5	36.0	50.0	50.0	50.0	34.3
Bentone SD-1	0.36	0.0	13.0	14.0	16.0	32.0	40.0	25.0	50.0	35.0	40.0	29.4
Bentone SD-2	0.37	14.0	14.0	30.0	24.5	32.0	8.0	9.0	52.0	32.0	20.0	24.6
Bentone SD-3	0.56	0.0	13.5	13.0	12.0	45.0	14.0	30.0	32.0	49.0	37.0	27.3
Benathix 1-4-1	0.25	18.0	36.0	39.0	27.0	14.0	11.0	13.0	52.0	49.0	48.0	32.1

Table 2. Laboratory Test Results

CLAY	MANUFACTURER	PROCESS	BASE CLAY	ORGANIC MODIFIER
Attapulgate	Engelhard Specialty Chemicals	None	Attapulgate	None
Bentonite	NL Baroid	None	Bentonite	None
Attatone J	Bentec	Dry	Attapulgate	Dimethyl di(hydrogenated tallow) ammonium chloride
Attatone I	Bentec	Dry	Attapulgate	Dimethyl di(hydrogenated tallow) ammonium chloride
BC 90	Bentec	Dry	Hectorite	Dimethyl di(hydrogenated tallow) ammonium chloride
Bondtone	NL Baroid	Dry	Bentonite	Methyl benzyl di(hydrogenated tallow) ammonium quat
Claytone APA	Southern Clay Products	Wet	Bentonite	Proprietary amine
Claytone 40	Southern Clay Products	Wet	Bentonite	Dimethyl di(hydrogenated tallow) ammonium quat
ZHT	NL Baroid	Dry	Bentonite	Methyl benzyl di(hydrogenated tallow) ammonium quat
ZHT Aberdeen	Bentec	Dry	Ca Bentonite	Dimethyl di(hydrogenated tallow) ammonium chloride
P-1	Bentec	Dry	Bentonite	Dimethyl di(hydrogenated tallow) ammonium chloride
P-11	Bentec	Dry	Bentonite	Dimethyl di(hydrogenated tallow) ammonium chloride
P-40	Silicate Technology Corp.	Dry	Bentonite	Dimethyl di(hydrogenated tallow) ammonium quat
PC-1	Bentec	Dry	Bentonite	Dimethyl di(hydrogenated tallow) ammonium chloride
PT-1	Bentec	Dry	Bentonite	Dimethyl di(hydrogenated tallow) ammonium chloride
Suspentone	NL Baroid	Dry	Attapulgate	Dimethyl di(hydrogenated tallow) ammonium quat
TS-55	NL Baroid	Dry	Bentonite	Methyl benzyl di(hydrogenated tallow) ammonium quat w/ polymer (elastomer)
Tixogel DS	United Catalyst	Wet	Bentonite	Methyl benzyl di(hydrogenated tallow) ammonium chloride
Tixogel EZ-100	United Catalyst	Wet	Bentonite	Dimethyl di(hydrogenated tallow) ammonium chloride
Tixogel GB	United Catalyst	Wet/Dry	Bentonite	Dimethyl di(hydrogenated tallow) ammonium chloride
Tixogel PE	United Catalyst	Wet	Bentonite	Methyl benzyl di(hydrogenated tallow) ammonium chloride
Tixogel SP	United Catalyst	Wet	Bentonite	Methyl benzyl di(hydrogenated tallow) ammonium chloride
Tixogel TE	United Catalyst	Wet	Bentonite	Dimethyl di(hydrogenated tallow) ammonium chloride
Tixogel VP	United Catalyst	Wet	Bentonite	Dimethyl di(hydrogenated tallow) ammonium chloride
Tixogel VZ	United Catalyst	Wet	Bentonite	Methyl benzyl di(hydrogenated tallow) ammonium chloride
Bentone 27	NL Chemicals	Wet	Hectorite	Methyl benzyl di(hydrogenated tallow) quat
Bentone 34	NL Chemicals	Wet	Bentonite	Dimethyl di(hydrogenated tallow) quat
Bentone 38	NL Chemicals	Wet	Hectorite	Dimethyl di(hydrogenated tallow) quat
Baragel 3000	NL Chemicals	Wet	Bentonite	Dimethyl di(hydrogenated tallow) quat
Bentone SD-1	NL Chemicals	Wet	Bentonite	Methyl benzyl di(hydrogenated tallow) quat
Bentone SD-2	NL Chemicals	Wet	Bentonite	Dimethyl benzyl (hydrogenated tallow) quat
Bentone SD-3	NL Chemicals	Wet	Hectorite	Methyl benzyl di(hydrogenated tallow) quat
Benathix 1-4-1	NL Chemicals	Wet	Bentonite	Dimethyl benzyl (hydrogenated tallow) quat

#### Disclaimer

The work described in this paper was not funded by the U.S. Environmental Protection Agency. The contents do not necessarily reflect the views of the Agency and no official endorsement should be inferred.