Notes and quotes

“Abstract: The Bureau of Mines, U.S. Department of the Interior, has developed a novel method of flocculation dewatering of phosphatic clay wastes using poly(ethylene oxide) as the flocculant. Research was conducted to determine whether ethylene oxide gas was present in the air in the vicinity of disposed waste materials which had been flocculated with poly(ethylene oxide). Samples of clay waste materials containing poly(ethylene oxide) were prepared in stoppered glass bottles in simulated disposal environments. Gaseous samples, removed over a 75-day period using an airtight syringe, were injected into a gas chromatograph that was capable of separating ethylene oxide from air. The presence of ethylene oxide gas was not detected in any sample. To determine possible degradation products of poly(ethylene oxide), the properties and reactions of ethylene oxide and its polymers were reviewed. Based upon the literature survey and experimental study, it was concluded that adverse environmental effects were not likely to result from the use of poly(ethylene oxide) for flocculating phosphatic clay waste products.”

“Conclusions: Ethylene oxide gas was not detected in systems that simulated disposal environments of clay wastes flocculated with PEO. This study substantiates degradation studies on poly(ethylene oxide) performed in other laboratories and reported in the literature. After consideration of the literature and experimental data, it was concluded that adverse environmental effects were not likely to result from the use of poly(ethylene oxide) for flocculating clay waste products.”


“Abstract: Standard biochem. tests on rats and mice showed that poly(oxyethylene) [25322-68-3] (mol. wt. 2.5 x 10^7) administered i.p. or by gastric gagage daily for periods up to six months showed low toxicity, no cumulative effects, and no effect on the central nervous system activity. For chronic ingestion of poly(oxyethylene) with drinking water the threshold dose was calculated to be 10 mg/kg daily and the subthreshold dose to be 10 mg/kg. The maximum permissible concentrations of poly(oxyethylene) in reservoir water were calculated to be 0.125, 0.1, and 0.02 mg/l, respectively, for polymers of the 2 x 10^7, 3 x 10^7, and 5 x 10^7 mol. wts.”


“Because the polymer is nontoxic and has a negligible environmental impact (other than through the physical processes described in this study) the polymer appears attractive for routine use.”

Additional references

Ecological fate and effects data on POLYOX polymer WSR N-750

1. Biodegradability summary: Although POLYOX™ Water-Soluble Resin (WSR) polymers would be expected to biodegrade very slowly, adsorption studies have shown that low concentrations of POLYOX WSR polymers in wastewater will be removed on biological solids rather than being discharged with the treated wastewater. Biodegradation screening tests with unacclimated domestic microorganisms show less than 5 percent biooxidation in the 20-day biochemical oxygen demand (BOD) test. Aquatic effects data indicate moderate toxicity to Daphnia magna and very little toxicity to fathead minnows and bacterial populations.
Ecological fate and effects test data are summarized in Table 1. These results show POLYOX WSR N-750 is resistant to rapid biodegradation, relatively nontoxic to bacterial populations and fathead minnows. The resin is moderately toxic to *Daphnia magna* as indicated by a 48-hour LC50 of 51.7 mg/L.

**Table 1: Ecological fate and effects data on POLYOX WSR N-750**

<table>
<thead>
<tr>
<th>Biodegradation</th>
<th></th>
</tr>
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<tbody>
<tr>
<td>Chemical oxygen demand (COD)</td>
<td>mgO₂/mg POLYOX WSR N-750</td>
</tr>
<tr>
<td>Biochemical oxygen demand (BOD)</td>
<td>20-day BOD value, % biooxidation</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Ecological Toxicity&lt;sup&gt;b&lt;/sup&gt;</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Bacterial inhibition, IC50, mg/L</td>
<td>&gt;5,000 96-hour</td>
</tr>
<tr>
<td>LC50 with fathead minnow, mg/L</td>
<td>&gt;5,000 48-hour</td>
</tr>
<tr>
<td>LC50 with <em>Daphnia magna</em> (95% CI) mg/L</td>
<td>51.7 (31-71)</td>
</tr>
</tbody>
</table>

<sup>a</sup> Measured by procedures published in Standard Methods for the Examination of Water and Wastewater. More details presented in the test procedure section.

<sup>b</sup> These tests were conducted by procedures published by EPA (600/4-85/013) except for the bacterial inhibition test which is a bacterial growth test. Reference provided in experimental section.

Since POLYOX WSR biodegrades very slowly, but is not highly toxic to aquatic life or biomass from treatment processes, the main concern is related to potential build up in an environmental compartment. Due to the coagulant adsorption properties of POLYOX WSR, it was expected to be removed from water solution on solids. However, the initial biomass contact/adsorption study failed to show significant removals.

The use of BOD dilution water in washing the activated sludge and preparing the final contact solution may have caused the POLYOX WSR to remain with the salts in the BOD water rather than being adsorbed on the biomass during the two-hour contact period. It is known that POLYOX WSR complexes with alkali metal ions, such as the sodium and potassium contained in the BOD dilution water. Complexed POLYOX WSR would have less tendency to be adsorbed on the biological solids. Washing the sludge with BOD water was done to reduce the background organics and the associated interferences with the Berger’s Reagent method for POLYOX WSR analysis (see test method details).

A second adsorption study was set up avoiding the BOD dilution water by simply collecting activated sludge from a sewage treatment plant and contacting POLYOX WSR concentrations of 3, 5, and 10 mg/L for about 16 hours in laboratory reactors under aeration. The filtered supernatant from these reactors indicated complete POLYOX WSR adsorption based on data using the Berger’s Reagent method. The background organics in this activated sludge sample was not a problem for the POLYOX WSR analysis.

**Conclusions:** These studies indicate POLYOX WSR N-750 is adsorbed on biomass at test concentrations of 3, 5, and 10 mg/L, rather than being discharged with the treated effluent. Environmental tests indicate POLYOX WSR N-750 is resistant to rapid biodegradation, but relatively nontoxic to fathead minnows and bacterial population and only moderately toxic to *Daphnia magna*.

**Biodegradation testing procedures:** Biodegradation measurements were obtained using procedures which generally follow the biochemical oxygen demand (BOD) method published in Standard Methods for the Examination of Water and Wastewater, 16th ed., American Public Health Association, Washington, D.C. (1985). Method changes involved test period extended to 20 days; reaeration, if needed, was accomplished by dividing the BOD bottle contents between two BOD bottles, sealing, shaking 20 times and returning to the original BOD bottle, reading oxygen level, resealing and returning to incubator. Discussion of these modifications appears in Price, et al., “Brine Shrimp Bioassay and Seawater BOD of Petrochemicals,” published in J. Water Poll. Control Fed., January 1974. The reported values represent the average of all test bottles.

**Bacterial inhibition:** The test material is evaluated at selected concentrations in a mixture containing buffer, nutrients, growth substrate and microorganisms. This mixture of one ml of a suspension of seed microorganisms, 24 ml of dilution water from the standard biochemical oxygen demand (BOD) test, 4 ml of stock buffer solution from the BOD test, 10 ml of a yeast extract/sodium acetate solution, and 4 ml of an aqueous solution of the test material is incubated in an 8-ounce, narrow-neck, round bottle for sixteen hours on a platform shaker at ambient temperature (22±2°C). Seeded control bottles are used to measure growth or turbidity generated during the sixteen hours without the test material. The bottles are stoppered with cotton plugs during shaking to avoid contamination.

The degree of inhibition can be assessed from measuring (optical density at 550 nm) the turbidity levels of the test material at various concentrations. The measured optical density values are calculated as a percentage of the seeded growth control system by this equation:

\[
\frac{\text{Optical Density of Test Concentration}}{\text{Optical Density of Seed Control}} \times 100 = \% \text{ of control Optical Density}
\]

The percent of control values are then plotted against the log of test sample concentration. The test concentration corresponding to 50% of the control is termed as 50% inhibition concentration (IC50). Further information concerning the test is available.

2. **Toxicity testing with fish: definitive 96-hour static acute bioassay (fish)**

The definitive tests were conducted by EPA-ASTM procedures using 20 fathead minnows per test concentration in a total volume of 0.75 liters. Temperature, fish survival, pH and dissolved oxygen levels were monitored during the test period of 96 hours. Minimal aeration was supplied when the dissolved oxygen was below about 4 mg/L. All NPDES samples were tested within six hours after collection without pH adjustment or solids removal. Treated process wastewaters from lab studies were refrigerated when immediate testing was not possible.

Dechlorinated municipal water was used in tests as dilution water and in experimental controls. This same water was used for maintaining the fathead minnow population. The incoming water was treated with activated carbon in a series of two 8-10 gallon filters yielding a chlorine-free dilution water with a pH very close to 7.0. Total hardness of this water measured 40 to 60 mg/L as CaCO₃. The carbon beds were changed as needed to maintain water quality.

The test fish, fathead minnows, were obtained from a laboratory culturing program. Size of the minnows ranged from 1.5 to 3 cm.

3. **Toxicity testing with Daphnia magna:** Test practices closely followed those recommended by the EPA Committee on Methods for Toxicity Tests with Aquatic Organisms (9,10) except replicate concentrations are not routinely used.

Planned by using other toxicity (fish) data or broad range-finding test, the definitive test is designed to provide a series of from 5-10 geometrically equidistant concentrations plus a control. The test is conducted in 250 mL beakers containing 200 mL of the test solution and 10 Daphnia. The Daphnia neonates (first instars) used in testing are less than 24 hours old, and are obtained by isolating gravid females for approximately 20 hours.

Dissolved oxygen and pH are determined initially and at 48 hours for all test concentrations and controls. Mortalities are recorded at 24 and 48 hours.

The Trimmed Spearman-Karber computer program was used to calculate the 48-hour LC50 and 95% confidence intervals.

River water was used in preparation of test solutions. This water is soft and its quality is sufficiently high that it can be used for maintaining long-term Daphnia cultures. The following analyses were obtained on the water:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
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<tbody>
<tr>
<td>Total Hardness</td>
<td>60 mg/L as CaCO₃</td>
</tr>
<tr>
<td>Total Alkalinity</td>
<td>38 mg/L as CaCO₃</td>
</tr>
<tr>
<td>pH</td>
<td>7.1</td>
</tr>
<tr>
<td>Conductivity</td>
<td>250 μhmhos/cm</td>
</tr>
</tbody>
</table>
Daphnia magna used in the bioassay came from a culture maintained in the laboratory for several years. This culture was initially stocked from the EPA laboratory at Duluth, Minn. The culture is maintained at 19°-23°C in glass beakers filled with river water. Culture transfers and feeding are performed three times a week. The transfer consists of placing 20-30 young Daphnia into each of three 600 ml beakers of river water. The laboratory-prepared food consists of pulverized trout food, yeast, and alfalfa powder.

4. Adsorption on biomass: (initial test)
The potential for removal of POLYOX WSR on biological solids was evaluated in a single laboratory contact study. Domestic activated sludge was washed with BOD dilution water, centrifuged and reslurried in BOD water at 1600 mg/L concentration. POLYOX WSR solution was added to two flasks containing one liter of the slurry at POLYOX WSR concentrations of 10 and 100 mg/L, respectively. One flask of slurry was maintained as a control. All the slurries were mixed on a shaker table for two hours prior to being centrifuged and filtered through 0.45 µm membrane filters. The POLYOX WSR concentrations were analyzed by the Berger's Reagent method which showed no significant removal of POLYOX WSR on the biological solids at the 10 mg/L test concentration. The higher dosage presented analysis problems.

(Second Test)
Due to the potential adverse impact of the sludge washing with BOD water, a second test series was set up using activated sludge without any washing or dilution. The activated sludge was divided among four 1.5 L laboratory reactors. One unit was maintained as a control, while the others received POLYOX WSR dosages of 3, 5, and 10 mg/L. The reactors were aerated for 16 hours prior to settling the solids and filtering the supernatant through membrane filters as specified in the analytical method.

References
4. EPA TSCA 40 CFR, Part 797 (7/87).

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