

# A REVIEW OF THE RADIOACTIVE AND THERMAL STABILITY OF LOW DENSITY POLYETHYLENE ENCAPSULATED NITRATE SALT WASTE

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## ABSTRACT

The Rocky Flats Plant (RFP) is considering polymer encapsulation as a stabilization technique for the treatment of several mixed waste streams. Specifically, a low-density polyethylene (LDPE) extrusion process is being developed for low-level nitrate salts, RFP's largest volume mixed waste. Major advantages of the polyethylene extrusion process, when compared to conventional solidification technologies, are improved durability and stability of the final waste form, and significant volume reduction.

Since the nitrate salt waste contains low levels of radioactive isotopes, the final waste form must be resistant to the ionizing radiation produced by these materials. RFP conducted a literature review to investigate the effects of ionizing radiation on polyethylene encapsulated nitrate salts. The results of this review indicate the salt waste does not have a sufficiently high specific activity to degrade the polyethylene waste form. Additionally, calculations indicate the waste will easily pass the most stringent of the current gas generation acceptance criteria in place at intended disposal sites.

Another major objective of this review was to evaluate the thermal stability of the nitrate salt/polyethylene mixture. Encapsulating an oxidizer in an organic could potentially result in a chemically reactive mixture. Experiments conducted at the New Mexico Institute of Mining and Technology to evaluate the issue of thermal stability demonstrated that no exothermal reaction hazards exist. This conclusion is supported by researchers affiliated with the Japanese nuclear power industry.

## RADIATIVE EFFECTS ON POLYETHYLENE WASTE FORMS - A REVIEW

### Introduction

The Rocky Flats Plant (RFP) is considering polymer encapsulation as a stabilization technique for the treatment of several mixed waste streams. The waste stream of primary interest is composed of the salts of various metals, including some metals that are land disposal restricted (LDR) under the Resource Conservation and Recovery Act (RCRA). The specific activity of the nitrate salts is approximately 300 to 500 picocuries per gram, making this a low level mixed waste stream. Because the dried salt fails leachability and dispersible solid requirements, additional treatment is required to convert this waste into a form acceptable for land disposal.

One of the issues concerning polymer encapsulated waste is the effect of ionizing radiation on the polymer. Exposure of polymers to ionizing radiation can have two major consequences: 1) degradation of the polymer, and 2) gas production. Degradation of the polymer can breach the impermeable barrier and erode the monolithic form, allowing migration of the hazardous constituents. Excess gas generation is prohibited at the final disposal site.

Since the RFP waste contains low levels ( $\approx 400$  picocuries per gram) of radioactive isotopes of Pu, U, and Am, the final waste form must be resistant to the ionizing radiation produced by these radioactive materials. The potential for degradation and hydrogen gas generation as a result of radiation-induced reactions is a function of the specific activity of the waste and the chemical and physical properties of the stabilizing matrix.

Another issue concerns fire or explosion hazards associated with this application of polymer encapsulation. Encapsulating sodium nitrate, an oxidizer, with polyethylene, an organic, could potentially result in a chemically reactive mixture. Such a mixture of fuel and oxidizer could burn if exposed

to sufficient thermal energy, with subsequent release of additional energy and gasses.

The major objectives of this review were to evaluate the radiolytic and thermal stability of the nitrate salt/polyethylene mixture.

### Radiation Chemistry of Polyethylene

Ionizing radiation by definition has sufficient energy to break chemical bonds by removing electrons from atoms it encounters. The major effects of radiation on PE are described by Charlesby (1):

1. The evolution of hydrogen and low molecular weight hydrocarbons.
2. The formation of C-C bonds between molecules (dimerization or crosslinking). These bonds convert the polymer into a type that is partly insoluble and infusible.
3. An increase in unsaturation. At low doses, the degree of unsaturation is proportional to the dose but eventually tends to a maximum value.
4. Destruction of crystallinity. The percentage of material present in the crystalline form at room temperature decreases and eventually disappears with increasing dose.
5. Color changes. In common with many other polymers, polyethylene acquires a yellow tinge upon irradiation.
6. Oxidative reactions, particularly near the surface, when the irradiation occurs in the presence of oxygen.

These effects can be readily accounted for through several chemical reactions. The chemical yields of these reactions are reported as "G values," which are defined as the number of chemical events per 100 eV of absorbed energy.

**Crosslinking:** It is thought that crosslinking occurs as a result of radiolytic-induced cleavage of a chemical bond. The free radicals produced then link together, forming a carbon-carbon double bond.

Due to the nature of the reactions involved in crosslinking, a molecule of hydrogen is produced for each crosslink. This is mainly due to the saturated nature of PE. With each carbon atom in PE having the maximum number of bonds initially, carbon-hydrogen bonds must be broken to form more carbon-carbon bonds (crosslinking). The hydrogen radicals produced from this bond cleavage then unite to produce a molecule of hydrogen. Crosslinking is thus related to gas production on a one to one basis. Reported G values for this reaction are about 2 to 2.5 (2,3).

**Chain Scission:** Chain scission results from the cleavage of a carbon-carbon bond. This lowers the overall size of the polymer molecules and produces a general degradation of mechanical properties. Because of this degradation of mechanical properties, chain scission is among the least desirable effects of polymer irradiation. However, chain scission is a minor reaction in PE, involved in less than 5% of the reactions. G values are reported to be 0.1 to 0.2 in linear PE.

**Changes in Saturation:** The reaction of interest occurs when trans-vinylene double bonds are formed. The mechanism is thought to be the removal of adjacent hydrogen atoms, followed by the formation of a double bond. As with crosslinking, a molecule of hydrogen is produced for each carbon-carbon double bond formed.

The yellow color seen in PE upon irradiation is caused by this reaction (1,2,4). The G value for this elimination reaction is about 1.8.

**Oxidation Reactions:** Oxidation reactions are free radical reactions that typically occur at the surface of the polymer, where oxygen is available. They are generally neglected in studies of the effects of radiation on bulk properties of the plastic.

Because decomposition of the nitrate salt could serve as a source of oxygen, some oxidation could take place in the interior of the monolith. Due to possible side reactions and the stability of intermediates, the yield of products for each step in the process is less than that for the previous step. The net result is the removal of gas rather than the generation of gas at low radiation doses (1,2,3). G values are reported to vary from about 10 for oxygen consumption to about 0.6 for carbon dioxide production.

Of the four reactions discussed above, crosslinking is considered to be the predominate effect. Increased unsaturation has little effect on the mechanical properties, but occurs with nearly the same yield as crosslinking. Each of these two reactions results in the production of hydrogen on a 1:1 basis. Chain scission is a deleterious but infrequent reaction, occurring at a rate of about 5% of the two main reactions. Oxidation is generally neglected, but could play a role that has yet to be quantified. The occurrence of each of these reactions is linearly dependent on the absorbed dose of radiation. If necessary, certain compounds can be added to scavenge free radicals and suppress all these reactions.

#### Effect of Absorbed Dose

From the above discussion, absorbed dose is clearly the dominant factor determining the effect of radiation on PE. Therefore, the suitability of PE for a given function is primarily dependent on the total absorbed dose of radiation in that environment (assuming PE is suitable for the function in a non-radioactive environment). The total absorbed dose at which PE can be used for nitrate salt encapsulation must be determined.

Charlesby (1) breaks down the effect of dose as follows:

1. At low doses (0-2 megarads for LDPE and 0-10 megarads for HDPE), the primary effect is an increase in branching. The solubility and melting point is relatively unaffected but the viscosity of the melt is increased.
2. At higher doses (a few to several hundred megarads for LDPE) a gel-like structure is seen. The gel forms a 3-dimensional crosslinked network and is both insoluble and infusible. Some crystallinity still exists and this controls the mechanical properties below the crystalline melting temperature. Above this temperature, the LDPE is rubber-like. The degree of crosslinking is about 0.1-5%.
3. At high doses a transition region is seen with crystallinity nearly eliminated and crosslinking controlling mechanical properties. This state is referred to as "cheese-like" in nature.
4. At very high doses the PE develops a glass-like structure. Crosslinking in this state exceeds 1 carbon atom in 10.

Based on Charlesby's descriptions, and from the point of view of mechanical properties, a dose of several hundred megarads would be acceptable. Laghari and Hammoud (5) list 10 megarads as a dose for which PE is nearly always usable as a dielectric. Van de Voorde and Restat (6) give the same number as a dose for which PE can be used in terms of its mechanical properties. Both give 100 megarads as a dose for which PE is nearly always useful. These numbers are again corroborated by Clough (7) and by Tipton (8). Schonbacher and Stolarz-Izycka (9) give 200 megarads as the maximum dose for which PE is recommended for use. Moriyama et al. (10) concluded that PE could be used to encapsulate waste at doses up to 1000 megarads. A rough agreement is thus found among these authors that little or no deterioration in PE is seen below 10 megarads and that PE is useful for most applications up to 100 megarads. Because the proposed use of PE to encapsulate nitrate salt waste does not require any exceptional mechanical properties, an upper limit of 100 megarads can be used. As discussed previously, the mechanical properties are important to maintain the monolithic final waste form and ensure the occlusion of the salt within the PE.

An equally important criterion from the standpoint of waste transportation and storage is gas generation rate. Actual gas generation rates are seldom reported. G values are more commonly reported because they provide a more general basis of comparison. Three studies were found that directly address the production of gas from irradiated PE. Tipton (8) reports a total of 38 ml/g of gas generated from PE exposed to 550 megarads, or 0.0691 ml/g-megarad. This converts to a G value of 3.0, which is in agreement with G values reported in TRUPACT-II SARP (11). Moriyama et al. (10) calculated a G value of 2.4 for hydrogen generation. Zerwekh (12), working at the Los Alamos National Laboratory, investigated gas generation from radiolytic attack of PE waste. In doing this, both LDPE and crosslinked HDPE were placed in gas-tight cylinders and spiked with Pu. Spiking levels were selected to simulate Pu concentrations in waste of 200, 100, and 20 g/barrel. This corresponds to activity levels of  $4X10^5$ ,  $2X10^5$ , and  $4X10^4$  nCi/g, respectively. To simulate 20 years of gas generation in 6 months, the activity was increased by a factor of 40. Zerwekh found that radiolytic attack of LDPE generated gas in significant amounts at only the two higher Pu



loadings. The lowest level of Pu contamination did not produce measurable quantities of gas in LDPE. The HDPE was found to be remarkably resistant to any gas generation. Calculations produce a G value of approximately 2 for LDPE, again in agreement with the review in the TRUPACT-II SARP for alpha radiation. The specific activity of the lower Pu loading (producing no significant gas generation) was about  $2 \times 10^{-3}$  Ci or about 2 billion picocuries/g. RFP salt averages around 400 picocuries/g.

While it may seem intuitively obvious at this point that the specific activity of RFP's nitrate salts is below levels of concern, it is valuable to compare this activity to various waste acceptance criteria. From the literature G values presented, gas generation from RFP waste can be calculated.

The assumptions for these calculations are as follows:

- specific activity of the nitrate salts: 400 picocuries/g
- 50% salt loading by weight
- specific gravity of salt: 2.0
- specific gravity of salt/PE waste form: 1.35
- barrel volume:  $1.98 \times 10^5$  cc
- weight of waste in each barrel:  $2.67 \times 10^5$  g
- G value: 4 events/100eV.
- All radiation is assumed to be gamma
- All radiation is assumed to be absorbed by the polyethylene

These assumptions provide a "worst case" scenario. In actuality, the majority of the radiation is alpha and would be absorbed by the salt rather than the PE. The small amount of gamma radiation present would probably not be completely absorbed by the PE. This would result in a much lower G value and, therefore, much lower gas generation rate.

An acceptable gas generation rate for transportation (as defined in the TRUPACT-II SARP (11)) is  $6.5 \times 10^{-7}$  moles/sec-drum. A calculation of the gas generation per drum of RFP waste yields  $6.5 \times 10^{-13}$  moles/sec-drum. This is less than one millionth the maximum acceptable rate. A related requirement gives a maximum heat generation of 7 watts per drum. RFP's expected waste stream is calculated to yield approximately  $1.6 \times 10^{-6}$  watts, or a factor of 4 million less than the criterion.

Since the Rocky Flats nitrate salt waste stream is low level, and thus destined for disposal at the Nevada Test Site (NTS), it is perhaps more appropriate to compare anticipated gas generation rates to NTS acceptance criteria. There are currently no specific criteria for hydrogen gas generation in low level waste. However, NTS requires that pressure in the waste package be less than 1.5 atm. Assuming a headspace of 5% of the package, this allows for a generation of 5 standard liters of gas. The specific activity of the salt is insufficient to generate this quantity of gas. The RFP waste form should easily pass the NTS criterion.

An NTS requirement based on concentration states that the level of flammable organics (including hydrogen) must be less than 500 ppm in the headspace. This criterion is expected to be met at less than 23 grams of fissile material in the drum. With 0.0009 g/drum expected in the final product of PE encapsulated nitrate salt, the activity of RFP nitrate salts is 26,000 times less than this criterion.

## THERMAL STABILITY OF POLYETHYLENE WASTE FORMS - A REVIEW

### Introduction

Because the incorporation of sodium nitrate waste in polyethylene is a fairly new technology, the volume of published materials on the thermal stability of the waste form is limited. Researchers from the Center for Explosives Technology Research (CETR) at the New Mexico Institute of Mining and Technology and the Japanese nuclear power industry have evaluated the safety of the proposed waste form. Their results are summarized below.

### CETR Test Results

The CETR conducted a rigorous investigation of the thermal stability of the proposed nitrate salt/polyethylene waste form (13). The Los Alamos Technology Office (LATO) contracted with the CETR to specifically address the issue of the thermal stability of this mixture. The objective of the study was to "assess the risk for slow self-heating chemical reaction, leading to runaway thermal decomposition, explosion, or detonation following the long term storage or accidental burning" of the waste form. CETR concluded that the polyethylene waste form will not self-heat, self-ignite, explode, or detonate under any conceivable circumstances. They further concluded that the "waste form is a non-explosive, safe material to produce and transport by rail or road."

CETR's evaluation of the explosion and fire hazards associated with the transportation and storage of the polyethylene waste form included the following tasks:

1. Computer modeling of the detonation characteristics of the waste form at several waste loadings and densities;
2. experimental determination of the detonable characteristics of the waste form at each waste loading and development of a preliminary classification for waste form transportation according to the United Nations recommended test and criteria;
3. experimental determination of the lowest temperature at which thermal decomposition occurs for each waste loading;
4. experimental determination of the time-to-explosion at four different temperatures;
5. evaluation of the hazards associated with the transport and storage of the polyethylene waste form based on the results of tasks 1-4 above.

Test results from each of these tasks are discussed in the following Subsections:

**Computer Modeling of Detonation Characteristics:** Computer modeling code was used to calculate detonation and constant volume explosion characteristics of various mixtures of sodium nitrate and polyethylene. The likelihood of stable detonation actually occurring was judged by comparing calculated values with those of other extremely insensitive energetic materials. The computer model indicated that the waste form is not likely to detonate at any ratio of sodium nitrate to polyethylene. The most likely mixtures to detonate would be those near stoichiometric, or approximately 86 wt% sodium nitrate. The computer model calculated for this mixture, however, indicated that no reaction will occur.

**Experimental Determination of Detonable Characteristics:** The test procedure used was a standardized Navy procedure for evaluating the shock sensitivity of explosives and propellants. All tests on the 88 wt% sodium nitrate mixture gave negative results. Detonation did not occur in any of the tests.

**Thermal Stability and Burning Characteristics:** CETR conducted several laboratory tests to evaluate whether the proposed waste form would internally self-heat due to exothermic decomposition, and whether the waste form would contribute to an accidental fire in transit or in storage. Experiments included differential scanning calorimetry (DSC), gas evolution, time-to-explosion, and Henkin-McGill one-liter and small-scale (pressurized) cook-off tests.

DSC tests conducted on the 88 wt% sodium nitrate mixture indicated no exotherm was observed at temperatures up to 450°C.

Gas evolution was evaluated by heating samples to various temperatures for 4 hours. Tests were run on sodium nitrate and polyethylene individually, and on mixtures at 88, 70, 50, and 25 wt% sodium nitrate. No gas evolution was measured in the majority of tests over the temperature range 150 to 300°C.

Time-to-explosion tests were performed up to 475°C, which is the maximum temperature available using a Henkin-McGill apparatus. The objective of these tests was to determine the critical or lowest temperature at which an explosive sample exhibits thermal runaway. No positive test was obtained for any sample, indicating that the critical temperature, if any, must be higher than 475°C for the samples tested.

Pressurized ignition tests were conducted to observe whether the waste form would burn more readily under elevated pressure. No significant combustion was observed.

Thermal runaway, or cook-off tests, were performed to measure the critical temperature of the 88 wt% sodium nitrate mixture. No self-heating reactions were indicated at temperatures up to 311°C, the maximum temperature attainable using the apparatus.

**Evaluation of Transportation and Storage Hazards:** CETR concluded, based on reported theoretical and experimental investigations, that the polyethylene waste form will not pose an explosion hazard in transport and storage. They stated, "...an effort to classify the material for transportation would result in the material being regarded as a non-explosive oxidizer, or a non-flammable fuel.

CETR evaluated possible threats to a truck or railway carriage container loaded with 10 to 50 tons of the polyethylene waste form. These potential threats included 1) collision with another vehicle or train, 2) a truck running into a ditch, or a railcar derailment, 3) fire in a tire or brake spreading to other flammable materials such as diesel fuel or the contents of fuel tank or nearby tank car loaded with flammable material, 4) impact of handgun or rifle bullets, field gun projectiles, or antitank (shaped charge) weapons fired at the load, or 5) saboteur's explosive charge placed and detonated in direct contact with one of the blocks. None of the above listed threats will initiate a large-scale explosion or detonation in the solid block or even in the most sensitive mixture, the 88 wt% sodium nitrate.

CETR evaluated collision or derailment impacts at velocities up to 100 meters/second. Results indicated that friction or impact will not generate temperatures high enough for ignition even if surface burning occurred. A small-scale fire

caused by collision or derailment will be unable to produce any effect other than melting of the PE, which would re-solidify upon cooling.

Large-scale fires in a truck or railcar were considered by CETR. A fuel fire will cause the PE/salt waste form to burn as long as it is supported by the burning fuel. However, when the fuel is consumed or extinguished, the waste form will self-extinguish. There is a possibility that melted PE will continue to burn, but it can be easily and safely extinguished by foam or water.

Bullet or warhead impact, and sabotage were investigated by CETR. Results indicated that no attempts at sabotage the investigators could conceive of would succeed in detonation. The CETR report stated that "For all practical purposes, the proposed waste form is non-detonable." Impact by a high-velocity shaped charge might result in localized burning, but will quickly self-extinguish.

### Japanese Nuclear Power Industry Test Results

Researchers affiliated with the nuclear power industry in Japan conducted a safety evaluation of simulated high-nitrate wastes incorporated in plastic (15). They do not specify which plastic is used in these tests, but other publications from the Japanese discuss the use of LDPE for immobilizing radioactive sodium nitrate (10,14,16).

The Japanese tested the sensitivity of a mixture of 90 wt% sodium nitrate/10 wt% plastic (highest potentially explosive ratio) to detonation impact, mechanical action, and thermal action. No reactions were noted in any of the detonation or mechanical sensitivity tests.

Thermal sensitivity was evaluated using several ignition tests. The auto-ignition temperature reported for the pure encapsulation plastic was 455°C, which is consistent with auto-ignition temperatures reported for pure LDPE by other researchers (19). The auto-ignition temperature recorded for the 90 wt% sodium nitrate mixture was 315°C. For comparison, the auto-ignition temperatures of some common materials are presented in Table I.

TABLE I  
Auto-Ignition Temperatures of Some Common Materials

Material	Auto-Ignition Temperature, °C	Reference
90%NaNO <sub>3</sub> /10% Plastic	315	(15)
Douglas Fir	390	(17)
Paper	229-238	(18)
LDPE	445	(19)
Sulfur	232	(20)
Octane	220	(21)

### Hughes Associates Hazard Analysis Study

Rocky Flats contracted Hughes Associates to conduct an evaluation of the relative fire hazards associated with the production-scale polyethylene extrusion process, the potential fire hazards to the process from other activities in the building, and the adequacy of existing fire sprinkler systems and structural systems in the production building (22). The results of the study indicated that the fire hazards associated



with the proposed process are common to industrial extruder processes and are not unique to the intended application. No major issues were identified.

### CONCLUSIONS

The literature review demonstrated that the polyethylene-encapsulated waste form will remain chemically and mechanically stable under any conceivable transportation and storage environment. The waste form will not generate significant quantities of gas as a result of radiation initiated chemical reactions, nor will the waste form degrade as a result of exposure to expected levels of ionizing radiation. In fact, the low levels of ionizing radiation in the waste are expected to improve, rather than degrade, the mechanical stability of the waste form.

With regard to thermal stability, independent studies have demonstrated that the waste form does not present a fire or explosion hazard, and will remain thermally stable under any conceivable circumstance. Additionally, storage of raw polyethylene does not pose a serious fire hazard because of its low flammability.

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