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WASTE INCINERATION AND IMMOBILIZATION FOR NUCLEAR FACILITIES

(April–September 1977)

Andrew J. Johnson

Leong Q. Fong

Chemistry Research and Development
PILOT PLANT DEVELOPMENT

Rockwell International

Energy Systems Group
Rocky Flats Plant
P.O. Box 464
Golden, Colorado 80401

U. S. DEPARTMENT OF ENERGY
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Chemistry Research and Development
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SUBJECT DESCRIPTORS

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Incinerators
Waste Management
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ENERGY SYSTEMS GROUP
ROCKY FLATS PLANT
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WASTE INCINERATION AND IMMOBILIZATION FOR NUCLEAR FACILITIES

(April–September 1977)

Andrew J. Johnson and Leong Q. Fong

Abstract. Fluidized bed incineration and waste immobilization processes are being developed to process the types of waste expected from nuclear facilities. An air classification system has been developed to separate tramp metal from shredded combustible solid waste prior to the waste being fed to a fluidized-bed pilot-plant incinerator. Used organic ion exchange resin with up to 55 percent water has been effectively burned in the fluidized bed incinerator. Various methods of feeding waste into the incinerator were investigated as alternatives to the present compression screw; an extrusion ram was found to suffer extensive damage from hard particles in tested waste. A bench-scale continuous waste immobilization process has been operated and has produced glass from incinerator residue and other types of waste materials.

SUMMARY

An incineration-waste immobilization system is being developed for processing waste generated at nuclear facilities. Fluidized bed incineration of the waste and a continuous immobilization process are used to effect a 120 to 1 volume reduction of solid waste and produce a durable and leach-resistant product.

An improved air classifier for removing tramp metal from solid waste has been in use during several weeks of operation of the pilot-plant fluidized bed incinerator. The air classifier, used in conjunction with hand sorting, was effective in removing metal objects that could potentially damage the feed screw. Incineration of ion exchange resin was accomplished using several different techniques for pretreatment and feeding of the resin into the incinerator. The most practical method was to

drain off standing water and feed the 45 to 55 percent water-content resin directly into the incinerator at a point below the fluidized bed surface.

Alternative methods for feeding waste into the incinerator were investigated as a possible replacement for the compressing screw feeder. An extrusion ram was tested with various types of solid combustible wastes handled in the incinerator; results showed a high-pressure ram is necessary and that extensive damage to the barrel and piston results from bits of tramp metal and fractured ion exchange resin.

The bench-scale vitrification reactor has been successfully operated, producing glass from incinerator residues and other production type wastes. A metallic vitrification chamber heated by coiled wire resistance elements was used during initial tests. Inconel® 690, which was used to fabricate the chamber, was the most corrosion-resistant material of those tested under operating conditions. This material has been in use long enough to determine several operating parameters.

Waste is free-formed into glass buttons on a rotating plate, which is positioned beneath an adjustable orifice. This orifice controls the drop size of molten glass exiting the reactor. Annealing and then cooling is accomplished using a furnace-jacketed, vertical-tube annealer and a vibratory cooling conveyor.

Two different methods for vitrifying high efficiency particulate air (HEPA) filters in the waste stream were used: (1) incineration of the entire filter and vitrifying the incinerator residue and (2) separating the filtering media from the frame, incinerating the

frame, and vitrifying the media directly. Both methods offer feasible approaches to successful vitrification of used HEPA filters.

INTRODUCTION

The large volume of combustible waste generated at nuclear facilities, along with attendant shipping and storage problems, led to the establishment of incineration projects at several Department of Energy sites. The primary goal has been the development of methods for reducing the weight and volume of combustible waste and producing a waste product that is safe for shipment and storage. The technique under development at the Rocky Flats Plant is fluidized bed incineration followed by conversion of incinerator residues into a vitrified product.

A 9 kg/hr pilot-plant fluidized bed incinerator (FBI) that had already been constructed at this plant was used for the program. Solid combustible wastes, generated in production operations, are burned in a primary fluidized bed of sodium carbonate. This burning process provides *in situ* neutralization of hydrochloric acid resulting from the large amount of polyvinyl chloride (PVC) plastics contained in the waste. Liquid wastes, including chlorinated hydrocarbons, are burned in a similar manner. The low temperature process (below 625 °C in both the primary bed and fluidized catalyst afterburner) eliminates the need for refractory lining in the system. Neutralization of acid gases in the primary bed and the use of sintered metal filters in the off-gas line negates the necessity of a liquid scrubber.

Immobilization methods are being investigated for converting incinerator residues into a form that meets the criteria for acceptance at a permanent repository. These criteria would ban the acceptance of respirable fines; residues from incineration are fine powders and would therefore need to be immobilized. Various methods of waste immobilization have been investigated at nuclear sites in this country and abroad. These methods have included cementing, incorporation in bitumen and plastic, calcination, and vitrification. The repository criteria on combustibles and gas generators would

favor cementing and vitrification, with vitrification being the more favored method. After laboratory-scale experiments with most of the immobilization methods, vitrification was selected as the immobilization method for the program at the Rocky Flats plant.

There are differences between the rad waste applied to this program and the high-level waste that has been the object of previous vitrification programs at other nuclear sites. The lower activity level and extremely low heat output of the waste considered at Rocky Flats, along with the difference in composition, permit changes in process methods and product form. A major objective is that the immobilization method be simple and relatively inexpensive for processing the large volume of waste in this low-level category. Another objective is that the amount of additives required to produce an acceptable glass product be kept low.

The work covered in this report is part of an ongoing effort to ensure that any method developed will be acceptable by potential users and will meet criteria for the handling and storage of rad wastes.

DISCUSSION

Fluidized Bed Incineration

Solid Waste Handling

The pilot-plant fluidized bed incinerator used in this work was described previously.¹ No major modifications have been made in the unit over the past six months. A flow diagram of the unit is shown in Figure 1.

The new air classifier, installed under the primary shredder, has been used during a number of incinerator runs on solid combustible waste generated in the plant production operations. Tramp metal content of the combustible waste varies with the location on the plant site of the waste collection drum. Most of the tramp metal consists of short lengths of pipe, nuts and bolts, wrenches, and scrap metal. Table 1 shows separation results achieved in four representative runs, each of about 100 hours duration.

FIGURE 1. Fluidized Bed Incineration Pilot Unit

TABLE 1. Tramp Metal Removal From Combustible Waste

Run Number	Metal in Feed (%)	Removal Efficiency		Metal Into Incinerator (%)
		Hand Sorting (%)	Air Classifier (%)	
1	6.8	58.9	35.3	5.8
2	11.1	64.3	28.3	7.4
3	9.6	47.0	38.0	15.0
4	6.8	67.9	20.4	11.7
Average	8.6	59.5	30.5	10.0

The first two runs involved general-production combustible waste; the waste burned in the last two runs included large quantities of shoe covers and coveralls. Snaps and buttons that remain attached to the shredded cloth, along with other small metal objects, were pneumatically conveyed to the feed hopper from where they were screw-fed into the incinerator. The tramp metal collected at the bottom of the fluidized bed and was easily drained out without disturbing the operation of the incinerator.

Extrusion Ram Waste Feeder

Development of alternative methods for feeding waste into the fluidized bed incinerator are continuing. The present compressing screw provides an efficient feeding method for general plant combustible solid waste. This screw, with its 5 to 1 compression ratio, has the ability to tolerate small pieces of tramp metal remaining in the waste after air classification; however, it will not convey non-compressible wastes such as ion exchange resin and shredded HEPA filters. These materials are handled by being mixed with general waste, which is then burned in small batches. It would be desirable, however, to burn these materials unmixed.

A simpler alternative appeared to be an extrusion type ram. It would provide an air-tight seal into the incinerator and would occupy approximately the same physical space as the screw. A ram feeder was assembled with a 570-mm-long steel barrel bored to 31.8 mm and with a 31.4-mm-diameter cylindrical piston. Waste was fed into a 150- X 30-mm charging port from a small hopper positioned above the charging port. A variety of shredded

TABLE 2. Ram Feeder Results on Various Wastes

Material	Extrusion Length (mm)	Extrusion Force (kN)	Extrusion Pressure (MPa)	Discharge Appearance	Feed Rate (kg/hr)
General Waste	210	<2.7	<3.4	Loosely compacted	0.8
	820	40 to 65	50 to 80	Moderately compacted	—
HEPA Filters	210	<2.7	<3.4	Loosely compacted	1.0
	820	52 to 65	65 to 80	Moderately compacted	—
Ion Exchange Resin	210	40 to 52	50 to 65	Moderately compacted	1.7
	820	90 to 115	115 to 150	Tightly compacted	—

waste material was tested, including general waste, HEPA filters, ion exchange resin, and mixtures of all of these.

Ram actuation was with a 30-ton hydraulic cylinder powered by a 248-W (1/3 hp) hydraulic pump; maximum pump rating was 70 MPa at 12 ml/min. Piston stroke of the ram was 370 mm with a 150-mm travel beyond the charging port. The barrel length remaining beyond the fully extended piston was 210 mm. A 610-mm straight barrel extension and a 30-mm reducing extension were also tested, thus increasing the extrusion length to 820 mm and 240 mm totals.

Results from the ram feeder tests are summarized in Table 2. Although all waste materials could be extruded, the required extrusion pressures were quite high and the feed rates were low. The data indicated the feed rate would have to increase by a factor of 5 to 10 times to obtain the 9 kg/hr rate needed by the pilot-plant fluidized bed incinerator. Manufacturer's literature shows that a 2.2 kW (3 hp) hydraulic pump capable of supplying 120 ml/min at 70 MPa might accomplish this.

Following completion of the feeding test, the piston and barrel were disassembled and inspected. The barrel base and piston surface showed extensive scoring and some gouging where hard feed material

had been trapped between the piston and the barrel bore. Ion exchange resin is a surprisingly abrasive material, especially the resin beads that are fractured during the ram feeding. This material or shredded nail bits from the HEPA filters could have caused the damage. Scoring of the bore and piston resulted in increasing the extrusion pressure necessary to move the waste through the barrel. The extent of this damage casts doubts upon the long-term operability of an extrusion ram feeder.

Ion Exchange Resin Handling

Organic ion exchange resins are used in a number of operations in nuclear facilities, chiefly in water treatment and in actinide separation. Crushing of the beads during water treatment operations, with generation of fines and concurrent increase in pressure drop, necessitates replacement of the resin. In actinide separation, crushing and radiolysis by the actinides result in the generation of fines and dictate periodic replacement of the resin. Both of these operations generate a waste stream that must be treated before any disposal can occur.

In the past, final immobilization treatment usually consisted of the incorporation of resin with cement or bitumen. The resin was drained before being added to the fixation media; the water content

usually varied between 45 and 55 percent in the drained resin. The presence of excess water (either by addition or from the resin) in the cementing operation has caused cracking and breaking of the cement product.² Total disintegration of cemented resin caused by swelling has also been observed when water has been added to the solidified material.³ Since cement cannot contain much over 35 percent resin, waste-volume reduction is negligible. Incorporation of bitumen is difficult because of the high water content of the resin and the reactions between resin and bitumen at high temperature. In addition, it was found that the asphalt product also tended to swell and disintegrate in water.

Incorporation of ion exchange resin in plastic is another method that has been tested.⁴ A good product could be obtained using a 50-50 mixture of resin and polyethylene, but a drying step for the resin was essential for the operation. Outgassing of resin-loaded polyethylene products by radiation-induced decomposition was roughly the same as reported for bitumen products. The outgassing and flammability of the products would make it extremely difficult to have these fixation methods accepted for processing the waste for shipment and storage in a permanent repository.

The organic ion exchange resins used at most nuclear facilities have a polydivinyl benzene or polyvinyl toluene base. These materials should readily burn; however, the high water content and characteristics of the resin beads complicate the incineration process. If burned in an incinerator with a grate, the resin melts and drops through the grate, mixing with the residues and complicating recovery of the transuranics. Atomization of resin into a combustion chamber results in excessive entrainment of unburned resin into the offgas system. The resin beads fracture into small particles as a result of the rapid heating of the material. Burning of the resin on a hearth plate is not efficient because of poor solid-to-gas contact. Agitation of the resin to expose it to the combustion air is the best way to burn it. Suspension of the resin beads in a fluidized bed probably achieves maximum exposure of the beads to oxidation and should avoid the extensive carbonization experienced in other types of incinerators.

Ion Exchange Resin Incineration

The waste resin, when received for disposal, is under standing water. Methods for moving the resin from the storage or holding container to the incinerator are being investigated. Resin can be slurried and pumped, but slurries containing less than 80 percent free water are difficult to convey. Various types of pumps were tested with resin that had been drained of free water, which is the condition desired for incinerator feeding. Progressing cavity pumps, double-diaphragm high-solids slurry pumps, and lobe pumps were tried, both on-site and in some manufacturers' test laboratories. None were satisfactory.

At this time, the best method appears to be the pumping of a resin slurry into an incinerator feed hopper that is fitted with a screen base and lower drain. The excess water drains out, and the drained resin is then screw fed into the incinerator using a straight screw. No preliminary drying step is needed before the resin, containing 45- to 55-percent water, is incinerated at approximately 600 °C in the fluidized bed incinerator.

The effect of high water content in the resin beads was noted and compared with two different feeding methods: (1) conveying into the fluidized bed beneath the surface and (2) conveying in at a higher level to allow the resin to fall onto the surface of the fluidized bed. If added above bed, an effect similar to corn popping occurs. The resin beads explode as a result of interstitial steam generation, and small fragments are carried out of the combustion zone before complete combustion is achieved. When the resin beads are introduced beneath the surface of the bed, however, premature resin carryover is eliminated, and combustion occurs in the bed media.

The waste resin that was actually burned in the tests was a sulfonated styrene-divinyl benzene copolymer that had been used for process water cleanup. After the resin was conveyed into the drain-equipped feed hopper, a vibrator was used on the hopper to prevent bridging of the damp resin. The vibration also was found to aid in the removal of free water from the resin. Feeding into the incinerator was by means of a straight screw, which conveyed resin to the lower part of the fluidized bed.

TABLE 3. Incineration Results on Ion Exchange Resin

Total Burned	1136 kg
Feed Rate	5.7 kg/hr
Waste Reduction	
By Weight	7:1
By Volume	8:1

Two week-long runs, averaging 100 hours each, have been conducted burning ion exchange resin. After each run, all incinerator residues were collected and weighed; composite samples were collected during the course of the run and were submitted for analysis. Results tabulated in Table 3 are combinations of both runs.

The waste resin had been used for water cleanup. This resulted in a considerable amount of inorganic salt in the residues; the major single component was sodium sulfate. The resin had enough heat content to make supplemental fuel unnecessary once the incinerator system attained steady state conditions. Further tests are planned after the installation of a variable compression screw, which is now being designed.

Waste Immobilization

Reactor Development

A bench-scale vitrification reactor has been designed, fabricated, installed, and has operated intermittently for the last six months. The vitrification reactor has a cylindrical metallic chamber (100 mm ID × 530 mm long) that is heated in an electrical resistance furnace; a schematic of the reactor is shown in Figure 2. The melting or dissolution section of the chamber is approximately 430 mm long and the fining section is 100 mm long. To ensure that the material in the fining section is a homogeneous molten glass, underflow and overflow baffles are located between the two sections. The molten glass flows from the melting section to the fining section and out through the discharge valve. Openings provided on the reactor chamber are for (1) feed input, (2) offgas exhaust, (3) product

FIGURE 2. Vitrification Chamber

discharge, and (4) control valve adjustment. The offgas exhaust and the control valve adjustment will be combined into one opening if tests show this is feasible. Minimizing the number of openings on the reactor chamber reduces heat loss with a corresponding decrease in the heat requirement.

Three semicylindrical heating elements surrounding the vitrification chamber provide approximately 3 kW of available energy. These electrical resistance elements are constructed with coiled wires of chromium-aluminum alloy embedded in a high-thermal-conductivity cement for 1200 °C operation. The reactor chamber and heating elements are completely insulated with 100-mm-thick ceramic blocks. These insulating blocks are composed of high-purity aluminum silicate fibers bonded with specially prepared binders suitable for temperatures of 1250 °C. The blocks are easily formed with a knife, which simplifies fabrication; a protective metal box encloses the insulation. The overall

dimensions of the vitrification reactor are approximately 0.6 m wide $\times$ 1.2 m long $\times$ 0.9 m high.

The vitrification process sequence is as follows:

1. Premix the feed, which consists of FBI ash, HEPA filter ash, tributyl phosphate (TBP) ash, mineral resin, diatomaceous earth filter aids, sodium carbonate, and boric acid.
2. Heat the vitrification reactor to 1050 °C.
3. Feed the mix into the reactor and heat in the reactor for 0.5 to 2 hours.
4. Adjust the product discharge valve to control the rate of molten glass dropping out of the reactor onto the rotating forming plate to produce free-formed glass buttons.
5. Anneal and cool the vitrified glass buttons.

The first and second metallic reactors were constructed from short lengths of Hastelloy® C and Inconel 600 pipes, respectively. Both materials were more readily available than Inconel 690, which appeared to have the best corrosion resistance in crucible tests.¹ These two reactors experienced severe corrosion within 40 hours at the 1050 °C operating temperature. The Hastelloy C reactor corroded through in areas above the melt line. Stainless steel pieces that accidentally fell into the Inconel 600 reactor formed a eutectic mixture with the Inconel metal, and caused the metal to corrode. The reactors operated long enough to substantiate the general principle of using a resistance heated metallic reactor in the immobilization process.

After operating the first two reactors, the following conclusions were made:

1. The chamber volume must be designed to compensate for the carbon content (heat available) in the glass mix. To reduce flame impingement, which causes hot spots on the reactor wall, the allowable heat release in the chamber should be less than 745 MJ/(hr m³).
2. To eliminate plugging in the feed tube, the tube must be enlarged to 50 mm in diameter for a feed rate of 7.5 kg/hr.

3. Excessive amounts of particulates or dust are carried out by the exhaust gas; thus, the gas velocity in the chamber should be reduced and an offgas cleaning system should be installed.
4. The restricted orifice used on the product discharge valve can be adjusted to control the drop size and rate of molten glass dripping out of the reactor. The valve is also “positive closed” without an additional mechanical device or cooling.

The third reactor was constructed of Inconel 690. This reactor operated at approximately 1100 °C for over 40 hours with no noticeable corrosion. The major problem with this and the first two reactors has been burn-out and electrical shorting of the electrical resistance heating elements. Continuous operation of the reactor at production temperatures causes the coiled wire elements to break. An electric resistance furnace, heated by silicon carbide glowbars, will replace the coiled wire elements. The glowbar furnace is capable of continuous operation at 1500 °C; however, it probably will not be operated above 1200 °C. Minor modifications to the furnace will be made so that the metal reactor chamber, including the outlets, are totally enclosed for efficient heat retention.

Glass Former

Among the various forming methods considered and tried were several cavity molds and a free-forming technique. Because of the complexity of a marble-forming machine, that technique was rejected as a forming method. The free-forming technique, which produces a button shaped product, is being favored because of the simplicity of equipment design and operation. The product discharge valve is a restricted orifice located on the bottom of the vitrification reactor. Drops of molten glass are fed out of the reactor onto a rotating metal disk, resulting in 13-mm-diameter by 5-mm-thick glass buttons. The forming plate also cools the glass to less than 600 °C to prevent buttons sticking to each other on contact. Initially the forming plate was constructed of aluminum but was replaced with an Inconel plate when it became warped from the heat.

Two continuous glass button annealers that automatically receive the buttons from the forming plate were tested. The first annealer, consisting of a horizontal screw conveyor jacketed by a tube furnace, tended to break or shear the buttons. The preferred annealer is a vertical metal tube jacketed by the tube furnace. It has an electro-magnetic vibratory feeder, slightly below the bottom of the vertical metal, which is filled with glass product buttons. The vibratory feeder controls the button residence time in the annealing furnace. The furnace operates at 500 °C.

The vibrator feeder used with the annealer also serves as the glass button cooler. While on the vibratory feeder trough, the buttons are cooled by means of ambient room air from 500 °C to 35 °C in approximately 12 minutes.

Product Integrity

Product integrity depends on the physical characteristics of the glass buttons. The ability of the glass button to withstand breakage during shipping was one of the most important considerations for long-term storage. Three tests considered as means of determining the glass strength were (1) impact, (2) abrasive grinding, and (3) compression.

The impact test is conducted by dropping a known weight onto the glass. From the distance of fall, the kinetic energy required to break the glass button can be calculated. In the abrasion test, a cylindrical container is filled with the glass buttons and is placed on a roller mill. The buttons tend to rub against each other as they would in a shipping container. This test provides a means of determining the resistance of the buttons to fracture or chipping. The other test is the compression test, during which a sphere is pressed onto the glass button and the load required to fracture is measured. All three methods are presently being evaluated. The compression test appears to be the simplest. Preliminary results indicate that the 13-mm-diameter buttons will withstand approximately 20.7 to 27.6 MPa compression before fracture.

Immobilization of HEPA Filter

Used high efficiency particulate air (HEPA) filters are in the waste stream of virtually all nuclear

facilities. Current practice for the disposal of used filters is either by compressing or sectioning the filters before packaging for shipment to an approved waste site for storage. Development work was directed towards immobilizing the HEPA filter in a glass product; two processing methods were considered for the HEPA filters. The first was to burn the filter in a fluidized bed incinerator and incorporate the residue with additives and other wastes to form glass in a vitrification reactor. The other method was to cut out and separate the filter media from the wooden frame and then process the media with glass additives to form glass.

The HEPA filter composition is basically a wooden frame (61 weight percent), filter media (10 weight percent), and separator material (29 weight percent). The filter frame material is an exterior wood particle board that is 0.75 inch thick and bonded with phenolic resin.⁵ In addition to fiberglass, the filter media contains 6 percent by weight of crocidolite fibrous blue asbestos. The separator material is crysolite asbestos (3 MgO·2 SiO₂·4 H₂O), which is formed into a water-repellant, corrugated, asbestos paper. Adhesives and sealants are used to seal the filter medium into the frame, to seal frame corners and edges, and to cement gaskets to the frame. The chemical composition of the fiberglass, crocidolite asbestos, and crysolite asbestos are given in Table 4.

Laboratory scale experiments were conducted to determine methods for vitrifying HEPA filter ash after the entire filter had been incinerated. A mix containing 70 percent HEPA ash, 20 percent B₂O₃, and 10 percent Na₂O produced a viscous, nonpourable melt at the 1050 °C operating temperature. A glass with good pouring characteristics could be produced, however, when a mix of 35 percent HEPA filter ash, 35 percent general FBI ash, 20 percent B₂O₃, and 10 percent Na₂O was heated to the 1050 °C temperature.

Tests were also made to determine if the filter media could be punched out of the filter frames and vitrified directly without a preliminary incineration step. The filter media and spacers were first converted to a powder by ball-milling and then were

TABLE 4. Composition of HEPA Filter Components

Chemical Composition	Fiberglass (wt %)	Crocidolite (wt %)	Crysolite (wt %)
SiO ₂	60	51.1	46.7
MgO	13	2.3	50.7
Al ₂ O ₃	21	—	0.2
CaO	6	—	—
FeO	—	35.8	0.8
Na ₂ O	—	6.9	—
H ₂ O	—	3.9	—
Fe ₂ O ₃	—	—	1.6
Total	100.0	100.0	100.0

mixed with various additives for testing. Heating the powdered HEPA media to 1050 °C without additives resulted in a non-molten hard cake. A mix of 70 percent powdered media with 20 percent B₂O₃ and 10 percent Na₂O was found to produce a melt that can be easily poured to form disks or buttons.

CONCLUSIONS

1. The air classifier for separating tramp metal from solid combustible waste has operated effectively during incinerator runs with actual plant production waste.
2. Use of an extrusion ram for a waste feeder on the FBI does not appear to be an effective alternative to the screw feeder.
3. Ion exchange resin can be burned efficiently when the resin is added beneath the surface of the fluidized bed.
4. An Inconel 690 metal reactor has proven to be resistant to corrosive effects encountered during production of glass from waste materials.

5. Replacement of coiled-wire resistance elements with glowbars for vitrification reactor heating is expected to significantly increase on-stream time.
6. Conversion of waste-stream HEPA filters to an immobilized form can be accomplished either by an initial incineration step before vitrification or by separating the media from the frame and subjecting the pulverized media to the vitrification process.

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