NASA TECH BRIEF

Ames Research Center

Halogenation of Microcapsule Walls

The incorporation of halogenated fire-extinguishing and fire-retarding additives into paints, coatings, sealants, and potting compounds is very desirable, but the number of materials which can be used is severely limited. High vapor pressure, toxicity, chemical reactivity and the fact that most of the additives are liquid are among the factors which limit use of the halogenated compounds.

An investigation was made of the possibility of microencapsulating fire-extinguishing and fire-retarding additives in order to minimize their deleterious properties and to alter the apparent physical and chemical properties of the additives. Microencapsulation is a process whereby microscopic portions of liquids or solids are individually coated with a layer of a polymeric film such as gelatin, nitrocellulose, or polyvinyl alcohol. Diameters of the capsules generally are of the order of 10^{-3} to 1 millimeter.

A variety of fire-retardant materials, primarily halogen compounds, were evaluated for modification in this manner. Current encapsulation technology requires mutual compatibility among the materials to be encapsulated, the encapsulants, and the solvents used. Unfortunately, the two candidate encapsulant materials meeting compatibility criteria, gelatin and gelatin-phenolic resins, are themselves good fuel, and a technique for halogenating these materials after capsule formation had to be developed to overcome this drawback.

The same general procedure is used for halogenation of the confining walls of both gelatin and gelatin-phenolic resin capsules: The capsules are carefully stirred into distilled water; a weighed amount of halogen is dissolved in the water and one mole of potassium halide per mole of halogen is added to aid dissolution of the halogen. The free halogen is allowed to react with the capsule wall material for a fixed length of time (about 2 hours). The capsules are then washed several times with distilled water, drained, and placed on trays to dry.

The halogenation is not reversible at ambient conditions, and halogen is not released from capsule walls at temperatures up to at least 60°C. The principal reaction taking place with gelatin is apparently a complexing of the amine groups with the halogen. Up to 11% bromine or up to 34% iodine has been introduced into wall material by varying the concentration of halogen in solution and the reaction time. Other significant factors are pH and temperature.

Halogen reacts (substitutes) at uncrosslinked sites of the phenolic resin in gelatin-phenolic resin wall material; the substitution is *ortho* and *para* to aromatic hydroxyl groups. The degree to which the phenolic resin is internally crosslinked and linked to the gelatin determines the number of sites available for halogen substitution. Amounts of iodine up to 55% or bromine up to 60% can be introduced into the wall material by controlling the following parameters: (1) phenolic resin-to-gelatin ratio; (2) crosslinking in the phenolic resin; (3) available halogen; (4) halogenation reaction time and temperature.

Usually a 10% halogen content will render the capsule wall nonburning; any higher content will enhance the flame-retardant properties of the selected internal phase material. Halogenation also tends to decrease the permeability of wall materials to the encapulated materials, especially at higher temperatures.

(continued overleaf)

Notes:

1. Alternative approaches to production of fire-retardant capsule walls for halogen compounds include overcoating of capsules with a second wall, and incorporation of fire-retardant materials into the primary wall during encapsulation.
2. This information should be of interest to manufacturers of encapsulants and similar gelatin materials where fire resistance is of importance, as in some types of fire-retardant paints.
3. The following documentation may be obtained from:

   National Technical Inforation Service
   Springfield, Virginia 22151
   Single document price $3.00
   (or microfiche $0.95)
   Reference:
   NASA CR-73374 (X69-19050), Encapsulation of Halogen Containing Compounds.

4. No additional documentation is available. Specific questions, however, may be directed to:

   Technology Utilization Officer
   Ames Research Center
   Moffett Field, California 94035
   Reference: B 72-10161

Patent status:

No patent action is contemplated by NASA.

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