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CC: A. C. Huiston
S. A. Salvage
T. N. Shipley
R. N. Taylor
File: 81-9

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TO: R. F. ANDERSON

FROM: R. G. ALSUP R. G. A.

DISPOSAL OF SOLID "TEFLON" WASTES CONTAINING C-8 APC

Introduction

Some of the solids wastes from the Fine Powder and Dispersion Area of the "Teflon" plant contain small amounts of toxic perfluorocarboxylic dispersing agents. A cheap and acceptable method of disposing of these solids is in a land fill, provided that the toxic agents are reduced to an acceptable level. Since the dispersing agents are volatile at moderate temperatures, we set out to determine the conditions necessary to bake the resins to a non-toxic state.

Summary and Conclusions:

1. The dispersing agent level of the solids waste stream is reduced to a quite acceptable level (below 2 ppm) by baking at 175°C for 5 hours.
2. Without a pretreatment, a small amount of the perfluorocarboxylic acid dispersing agent would be leached into the ground water.

Discussion:

A system for collecting the solid wastes from the Dispersion and Fine Powder Area was proposed by Anderson and Chren. This involved the use of sumps and settling basins to concentrate the solids. Solids in the form of coagulum from wax filters and wax separator settlings were also to be collected in this system. A flow sheet of the proposed system is attached. The total amount of solids is estimated at ca. 768 pounds per day at current operating conditions.

Samples of solid wastes were obtained from the collection points in the proposed system. These samples were representative of material to be obtained from the fine powder sump, the wax trap, the bottom of the wax separator and the sump from the Dispersion Area. The make-up of these samples was as follows:

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- Fine Powder Sump - This material looks like contaminated, wet, fine powder. The material contained 36.2% water and 130 ppm perfluorocarbon dispersing agent calculated as C-8 APFO.
- Wax Trap - appears like wax. Lost 8% by weight on heating to 175°C. Contains ca. 30 ppm C-8.
- Bottom of Wax Separator - appears like wet fine powder and contains about 65% water and 240 ppm C-8.
- Dispersion Area Sump - This sample was actually obtained from the bottom of the stabilization tanks and was a mixture of a 60% dispersion liquid and a solid containing about 30% water. The material contained ca. 300 ppm water. In most of the work done with this mixture, both the liquid and solid phase were analysed.

1. Leaching

As a first step, it was determined that the dispersing agent could be leached from these solids. This was done by adding 100 grams of the sample from the different sources to 100 ml of water in a Waring Blendor. After blending for 5 minutes, the water was filtered and analysed for dispersing agent by the titration method (see below). The data shown in Table I show that the C-8 dispersing agent could indeed be extracted by water. This is the conclusion reached by Crandell in 1964 (see attachment).

Table I

The Extraction of C-8 Dispersing Agent
from "Teflon" Samples in Water

<u>Sample Source</u>	<u>PPM C-8 Extracted</u>	
	<u>1st Extraction</u>	<u>2nd Extraction</u>
Fine Powder Sump	30	19
Wax Separator	103	15
Wax Trap	39	17

2. Baking

The samples were baked at 175°C for 3, 5 and 20 hours. Analysis of these samples showed almost complete removal of the dispersing agent at 5 or 20 hours; after 3 hours treatment only the sample from the fine powder sump contained any residual C-8.

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TABLE II

ANALYSIS OF PERIFLUOROCARBON DISPERSING AGENT
IN "TEFLON" SOLIDS WASTE

<u>Sample Source</u>	<u>Hours Heated @ 175°C</u>			
	<u>0</u>	<u>3</u>	<u>5</u>	<u>20</u>
Fine Powder Sump	130	100	2.7	0.24
Bottom of Wax Trap	240	<10	2.4	----
Wax Trap #8	(30)	0	0.73	----
Stabilization Tank				
Liquid	460	0	1.1	0.20
Solid	130	<40	0.77	0.41

APFC. The temperature of 175°C was chosen as one easily reached by most ovens and because it is below the flash point of the wax used (205°C).

3. Analysis

Two methods of analysis were used for the determination of perfluorocarbon dispersing agents in the waste streams. The procedures are included here for completeness. One made use of the property that perfluorocarbon dispersing agents can be distilled with water from a strongly acidified slurry. This distillate can then be titrated to determine the amount of acid (as perfluorooctanoic acid ($C_7F_{15}CO_2H$ — MW 431). In these studies the following procedure was followed:

1. A 100 gram sample (+200 ml of distilled water) was cut in a Waring Blendor. The slurry was added to a 500-ml flask and 25 ml of concentrated phosphoric acid added.
2. The flask was fitted with a distilling head and condenser. The slurry was heated to boiling and the condensate collected in an Erlenmeyer flask containing 50-ml of distilled water that had been carefully neutralized to a pink end-point with phenolphthalein indicator. Distillate samples were collected each 5 minutes and titrated with 1/100 N sodium hydroxide.
3. When the titrations indicated that no additional acid was being distilled (ca. 3 titrations or 15 minutes) the titre was added and the amount of dispersing agent calculated according to the following.

$$\text{ppm C-8} = \text{ml} \times 43.1$$

This method checked very well with known spiked amounts of C-8 in the slurry.

The other method is based on the formation of a complex between methylene blue and the dispersing agent. While methylene blue is not extracted from acid solution into chloroform, the complex is partitioned into the chloroform layer. The intensity of the coloration in the chloroform layer is related to the concentration of dispersing agent in the aqueous phase. Nonionic dispersing agents, such as "Triton" X-100, do not interfere. The method is sensitive to 0.05 ppm dispersing agent in the aqueous phase.

In this method, the following procedure was used:

1. The sample was prepared (as in the method above) by cutting a 100 gram sample in a Waring Blendor,

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with water, acidifying with H_3PO_4 and distilling..
For this test, 500-ml of distillate was collected..

2. A measured portion of the distillate was added to a 100 ml volumetric flask and diluted with water.. The measured amount depended on the amount of C-8 in the distillate. For instance, in the baked samples, a 25 ml portion, diluted to 100 ml, was used; for the as-received sample from the stabilization tank, a 0.2 ml sample, diluted to 100-ml was used.
3. The 100-ml from the dilution of step 2 was added to a 250-ml separating funnel and 1.0 ml of 1-9 HCl solution, 1.0 ml of methylene blue solution (0.500 gms/liter), and 10.0 ml of chloroform were added. The mixture was shaken vigorously for 2 minutes, allowed to settle and the chloroform layer withdrawn.
4. The chloroform layer was analysed in a spectrophotometer in a 11-cm glass cell by scanning from 700 m μ to 600 m μ using chloroform in the reference cell. The peak absorbance for the sample at 650 m μ was measured.
5. For these studies, it had been shown that 0.0057 units on the spectrophotometer = 1×10^{-6} grams of C-8. From the amount of absorbance and the factor from step 2, the amount of C-8 in the sample was calculated.

The two methods checked within 10% for the wax separator sample.

RGA:sc
Attachment