27
7-9-70
25 to UTIS

UCID- 17136

Lawrence Livermore Laboratory

A RECLAIMING PROCESS FOR SCRAP DEUTERATED POLYETHYLENE (CD_2)

Leonard E. Caley

May 20, 1976

This is an informal report intended primarily for internal or limited external distribution. The opinions and conclusions stated are those of the author and may or may not be those of the laboratory.

Prepared for U.S. Energy Research & Development Administration under contract No. W-7405-Eng-48.

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

A RECLAIMING PROCESS FOR SCRAP DEUTERATED POLYETHYLENE (CD₂)

ABSTRACT

A laboratory version of a process to clean contaminated deuterated polyethylene (CD₂) was modified slightly and scaled up to produce 2 lb of clean, reusable CD₂ polymer.

A complete cycle of the process is:

- Melt the CD₂ polymer at 30 vol% in xylene at 105-110°C.
- Pressure filter the hot solution at 103 to 138 kPa (15-20 psi).
- Cool the solution to precipitate the polymer and separate it from the liquor.
- Vacuum dry the precipitate at about 266 Pa and 37°C.

INTRODUCTION

Three conditions stimulated interest in recovering scrap CD₂ polymer using the process developed by LLL during the first half of 1973:* 1) increased Laboratory demand for CD₂, 2) a short supply of CD₂, as revealed in an inventory taken by Phil Fleming in July 9, 1974, and 3) the high cost of setting up a pilot plant to make virgin polymer.

NOTICE
This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Energy Research and Development Administration, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

* L. P. Althouse, Recovery of (Dirty) Scrap CD₂ by the Solution Precipitation Technique, Lawrence Livermore Laboratory, Chemistry Dept. Technical Note No. 73-34(1973). Readers outside the Laboratory who desire further information on LLL internal documents should address their inquiries to the Technical Information Department, Lawrence Livermore, Laboratory, Livermore, CA 94550.

RECOVERY PROCESS

The scrap CD_2 polymer was added to xylene at 30 vol%, heated to 105-110°C, and stirred until the polymer was completely in solutions (Phase 1, Fig.1).

The hot solution was transferred manually to the preheated pressure filtration unit (120 - 125°C) (Phase 2, Fig. 1.).

An inline filter (cartridge type) was separated from the pressure vessel by a valve which, after the solution was under pressure [103 to 138 kPa (15 to 20 psi)], was opened allowing the solution to be forced through the filter into the 5000-ml glass receiver container.

Precipitation of the CD_2 polymer takes place as the solution is cooled to below 60°C and eventually is separated from the liquor by vacuum filtration through Whatman No. 4 Filter paper.

The filter cake was then broken up and spread out in a thin layer in a tray for drying. Drying was achieved by vacuum oven technique at about 266 Pa and 37°C for 72 hr.

EQUIPMENT

The solution was made in two 2000-ml round bottom flasks heated in fitted mantels controlled by power rheostats. Stirring was done by magnetic stirring technique. Reflux condensers were used to control the hot vapors coming off of the solution during the process of melting the CD_2 in the xylene. Glass thermometers suspended down the center of the condensers measured the solution temperatures.

The solution was transferred from the flasks to the steam-heated pressure vessel that is pressurized to force the solution through the filter into the 5-liter container.

The precipitate was separated from the liquor by using a Buchner funnel with Whatman No. 4 filter paper.

The filter cake was broken up into fine powder and vacuum dried in a vacuum oven.

Analysis of the end product was the final step to determine the degree of cleanliness achieved by the process. Tables 1, 2, and 3 show the results of analyses run on 10 lots of cleaned CD_2 .

Fig. 1. Phase 1: Letters A through F represent the equipment used to dissolve the CD_2 : A. Condenser. B. Thermometer. C. Flask, round bottom, 2000 ml. D. Heating mantle. E. Magnetic stirrer. F. Power rheostat. Phase 2: Letters G through N represent the pressure filtration system equipment: G. Pressure bottle of dry nitrogen. H. Heater tape for heating pressure tank. I. Pressure tank for preheating dry nitrogen. J. Power state used to control the heat of heater tape. K. Pressure vessel for pressurizing the solution prior to forcing it through the filter. L. Valve. M. Filter (cartridge type). N. Receiver, glass, 5000 ml. O. Steam lines supply and return.

Table 1. Chemical analysis.

Specimen	C (wt%)	D (wt%)	H (wt%)	O (wt% by difference)
1	74.82	23.64	0.83	0.71
2	75.13	24.03	0.59	0.25
6	75.16	23.69	0.69	0.46
10	75.67	23.13	1.08	0.12

Table 2. Spectrochemical analysis for impurity elements (PPM).

Specimen	Si	Cu	Fe	Ag	Ca	Ti	Mg
1	10	5			10		3
2	200	50	30	30	20	10	
6	200	30	30	30	20	<10	
10	100	50	30	5	20	<10	

The following elements were not detected:

For specimen No. 1	<100	As, Ce, P, Na, Ng, Sb, V
	<30	Ba, Bi, Cd, Cr, Ge, Mo, Pb, Sn, Zn
	<10	Al, B, Co, Fe, In, Ni, Ti, Zr
	<3	Ga, Mn, St.
	<1	Ag, Be,
For Specimens Nos. 2, 6, and 10	<100	Na, NG, SG, V
	<30	Ba, Bi, Cd, Cr, Mo, PG, Sn, Zn
	<10	Al, B, Co, Ni, Zr
	<3	Ma
	<	Be, Mg

Table 3. Densities (measured by immersion method).

Batch No.	Density (g/cc)
1	1.078
2	1.064
3	1.064
4	1.062
5	1.063
6	1.060
7	1.064
8	1.064
9	1.063
10	1.062
Average density = 1.064 g/cc	

"Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Energy Research & Development Administration to the exclusion of others that may be suitable."

NOTICE

"This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Energy Research & Development Administration, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately-owned rights."

Printed in the United States of America
Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161
Price: Printed Copy \$; Microfiche \$2.25

<u>Page Range</u>	<u>Domestic Price</u>	<u>Page Range</u>	<u>Domestic Price</u>
001-025	\$ 3.50	326-350	10.00
026-050	4.00	351-375	10.50
051-075	4.50	376-400	10.75
076-100	5.00	401-425	11.00
101-125	5.25	426-450	11.75
126-150	5.50	451-475	12.00
151-175	6.00	476-500	12.50
176-200	7.50	501-525	12.75
201-225	7.75	526-550	13.00
226-250	8.00	551-575	13.50
251-275	9.00	576-600	13.75
276-300	9.25	601-up	*
301-325	9.75		

* Add \$2.50 for each additional 100 page increment from 601 to 1,000 pages:
add \$4.50 for each additional 100 page increment over 1,000 pages.

Technical Information Department

LAWRENCE LIVERMORE LABORATORY

University of California | Livermore, California | 94550