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# THIN FILM EVAPORATION FOR REUSE/RECYCLE OF WASTE ORGANIC SOLUTIONS

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## ABSTRACT

A thin film evaporator TFE has been used at PGDP to evaluate the feasibility of recovering waste organic solutions as reusable products or to reduce the waste disposal volume. The setup of the TFE and the hot oil heating system has allowed a wide range of solutions to be tested. These solutions include 1,1,1 trichloroethane, trichloroethylene, waste deplating solution, lacquer thinner (paint waste), and a polychlorinated biphenyls (PCB) and uranium contaminated waste oil solution. The recovery rate or waste volume reduction are presented for each solution tested. Cost savings for specific compounds and requirements for the use of recovered trichloroethylene and 1,1,1 trichloroethane are explained. Actual plant reuse of the recovered chlorinated solvents and the lacquer thinner has occurred.

## EXECUTIVE SUMMARY

The thin film evaporator (TFE) is being used to evaluate the feasibility of recovering waste organic solutions as reusable products or to reduce their waste disposal volume. The TFE has successfully recovered trichloroethylene, 1,1,1 trichloroethane and lacquer thinner. These chemicals have been returned to a large vapor degreaser, small parts cleaning bath in the pump shop, and the paint shop for field use testing. No problems have been found with the recovered products compared to the virgin solvents.

Testing of PCB (polychlorinated biphenyls) and uranium co-contaminated waste yielded a 28% decrease in disposal volume. The condensate recovered from the evaporation was below 50 ppm PCB, the federally regulated guideline. These test results proved volume reduction of PCB laden waste can be achieved prior to final disposal (incineration).

The technology is applicable to other sites for reduction of hazardous and contaminated waste feeds prior to incineration.

### Material Tested To-Date

Materials tested to-date include trichloroethylene, 1,1,1 trichloroethane, nickel stripper solution, co-contaminated oil, and lacquer thinner. Percent solvent recovery is defined as weight of solvent recovered divided by the weight of solvent processed. Maximum recoveries achieved were 96% for trichloroethylene and 98% for 1,1,1 trichloroethane. Recovery rates were found to be a function of:

1. Solids loading
2. Number of passes through the TFE system
3. Percent oil in the waste

Uranium removal has averaged 99% with typical 25 ppm uranium starting levels being concentrated to 350 ppm in the bottoms and the product reduced to 0.02 ppm.

The readdition of stabilizers to the recovered product suppresses the generation and accumulation of HCl during reuse of the

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solvent. This prevents corrosive reactions in the steel storage drums which would lead to pinhole leaks. To prevent corrosion, each specific solvent is inhibited with an acid accepting stabilizer. The level of inhibition of a solvent is measured by the non-amine acid acceptance (NAA). The average operating range of the NAA in the degreaser is 0.10 to 0.13% as NaOH. The maximum amount of inhibitor is needed in waste solvent after processing due to the interaction of the used solvent and the oil from dirty equipment. 1,1,1 trichloroethane is stabilized by 1,2 butylene oxide and secondary butyl alcohol. Trichloroethylene is stabilized by 1,2 butylene oxide, secondary butyl alcohol, cyclohexane oxide and diisopropyl amine.

The nickel-stripping solution is composed of two parts. The "A" component is a dry powder composed of sodium meta nitro benzyl sulfonic acid. The "B" component contains 2% ammonia and 20% aliphatic amine. TFE treatment of the nickel-stripper solution has yielded two distinct fractions, an ammonia-water mixture in the distillate and the Metex solid with high boiling liquids in the bottoms. The volume reduction achieved by separation of the ammonia-water mixture from the feed material was 55%.

The co-contaminated waste oil solution was high in suspended solids (75 g/l) and was filtered to 100-micron particle size prior to TFE processing. The PCB level of the initial sample prior to filtration was 6700 ppm. The TFE concentrated the waste in the bottoms solution to 55 g/l solids content. After processing, the bottoms concentration of 5200 ppm PCB's was lower than expected due to the solids removed in the filtration. The solids filtered out had PCB's sorbed to the solid material and produced the lower than expected bottoms concentration. The condensate contained less than 50 ppm PCB, which is below the federally regulated guideline for PCB's. The volume of the riginal solution contaminated with PCB's was reduced by 28% on a single pass through the TFE. Increased volume reduction would have been obtained if a lower initial suspended solids concentration had been present. A 50% recovery of the available volatile and water fraction of the solution was obtained. This proves volume reduction of mixed volatile wastes containing PCB can be achieved prior to final disposal (incineration).

**Test Materials Chemical Parameters**

The specifications for chlorinated solvents were taken from the manufacturers data sheets and our material specification. Additional parameters of interest were added, such as uranium and total suspended solids. The list of test parameters for the chlorinated waste is shown in Table 1.

Testing of the waste nickel stripper solution was monitored for percent volume decrease. Earlier testing in the laboratory showed the liquid fraction of the two-part, nickel-stripping compound was the active ingredient. The "B" liquid fraction is partially fractionated in the evaporation process. The high boiling liquid in the bottoms fraction could serve as an activator for the stripping process; however, the bottoms does have high suspended solids and separating the liquid was difficult. Recovery of the bottoms liquid for reuse was not feasible. The dried bottoms residue was ineffective as an additive to rejuvenate the stripping solution. The deplating operation would be affected by increased nickel concentrations from additions of the dried bottoms and would reduce the life of the bath rather than extend it. The condensate (product) yields an ammonia-water mixture that was tested in the laboratory and would not deplate nickel plated steel samples. The use of the ammonia-water mixture as a fertilizer is a reuse application of the condensate solution.

The waste oil solution contaminated with PCB's and U was analyzed for the characteristics listed in Table 2.

The lacquer thinner material was tested for the same general properties as the chlorinated solvents. The lacquer thinner waste solution produced a very clear condensate solution. The waste was identified by GC/MS comparison with stores current product of lacquer thinner. The test run recovered 57% of the total waste solution on the first pass. The solution was prefiltered and removed the paint fines that would have coated the TFE jacket walls and reduced heat transfer.

**Flow Diagram of the TFE**

The flow diagram of the thin film evaporator system is shown in Figure 1. This system includes an oil heater unit to provide heating of the jacketed evaporator section. The upper temperature limit of the hot oil heating system is 550°F. The evaporator is composed of a rotating center section, producing a thin film on the inside wall of the evaporator jacket, a condenser, feed pump, condensate pump, and bottoms pump.

**Optimum Machine Conditions**

The major parameters of operation that were monitored on all testing runs are listed in Table 3. Amperage load on the rotating center shaft ranged 62 - 67% of full load during all the testing. Increases in the vacuum on the TFE produced quicker product rates. The vacuum setting in processing the chlorinated solvents was five psia. At vacuum conditions above 13.5 psia, the rotating center section will vibrate. The vacuum can be used to draw feed material into the TFE to aid in priming the feed pumps provided the back pressure valve is set low enough to allow this to occur. Optimum conditions for processing the waste tested are listed on Table 4.

The feed rate will vary depending on the solids loading, percentage of oil in the solvent, and type of pump used. All trichloroethylene waste was processed in a single pass. The temperature difference between 1,1,1 trichloroethane and trichloroethylene represents the added heat required to process the higher boiling trichloroethylene. The flow rate will dictate how many passes the solvent should make before all available material is recovered. The time required to process a 55-gallon drum of waste chlorinated solvent was 4 - 6 hours depending on the percent solvent in the feed.

The co-contaminated solution was filtered prior to treatment. The TFE removed excess water and volatile organics from the waste oil mixture. High solids loading resulted in pumping out the bottoms area frequently.

The lacquer thinner solution processed through the TFE was filtered to 30 microns prior to treatment to remove paint particles from the solution. Solution fed very smoothly after the pump was primed and the correct pump feed pressure was established. The temperature in the vapor phase was still too low for single pass complete recovery.

The product cycle time has been plotted in control chart form for each of the materials tested and out of control points can be traced to changes in the feed pump settings.

## DISCLAIMER

expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

## TABLE 1
## TEST PARAMETERS FOR CHLORINATED WASTE SOLUTIONS UNDER SPECIFICATIONS FOR MATERIALS

| **Trichloroethylene** | **1.1.1 Trichloroethane** |
| --- | --- |
| Acidity | Acidity |
| Acidity after oxidation test | Flash point |
| Alkalinity | Fire point |
| Residue on evaporation | Non-volatile materials |
| Moisture | Water |
| Free halogen | Odor |
| Color | Color |
| Copper corrosion | Boiling range |
| Boiling range | Specific gravity |
| Specific gravity | |

**Additional Experimental Test Parameters**

Components
Uranium
Total suspended solids
Spectrochemical

## TABLE 2

## CONTAMINATED OIL SOLUTION ANALYSIS PARAMETERS

PCB
Uranium
Spectrochemical
Specific gravity
Water
Color
Residue after evaporation
Total suspended solids
Acidity
Boiling range

## TABLE 3

## PARAMETERS MONITORED FOR TESTING OF THE THIN FILM EVAPORATOR

Hot oil heater use time

Heater temperature control (maximum set point)

TFE temperature control (maximum set point)

Process temperatures

Point 1 overhead vapor temperature
2 fed in temperature
3 hot oil inlet temperature
4 hot oil outlet temperature
5 condenser outlet temperature
6 bottoms pump outlet temperature

Systems' Vacuum

Amperage load on the rotor

Product (condensate) cycle time

Feed pump setting

## TABLE 4

OPTIMUM MACHINE CONDITIONS FOR TFE TREATED WASTE

| | Jacket Temperature °F | Vapor Temperature °F | Vacuum psia | Product Cycle min:sec |
|---|---|---|---|---|
| Trichloroethylene | 315 | 183 | 5 | 1:48 |
| Trichloroethane | 250 | 163 | 5 | 1:26 |
| Cocontaminated Oil | 350 | 181 | 10 | 3:00 |
| Nickel Stripper | 317 | 212 | 0.5-21.5 | 3:15 |
| Lacquer Thinner | 355 | 210 | 10 | 1:47 |

# FLOW DIAGRAM OF THE THIN FILM EVAPORATOR SYSTEM

FIGURE 1