

Evaluation of Treatment Technologies for Listed Petroleum Refinery Wastes

HEALTH AND ENVIRONMENTAL SCIENCES DEPARTMENT
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Evaluation of Treatment Technologies for Listed Petroleum Refinery Wastes

Final Report

Health and Environmental Sciences Department

API PUBLICATION NUMBER 4465

**FIELD STUDIES CONDUCTED BY
THE API WASTE TECHNOLOGY TASK FORCE**

**REPORT PREPARED BY
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DECEMBER 1987

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PREFACE

Provisions for the land disposal ban of the 1984 RCRA Amendments, require EPA to develop best-demonstrated available technology (BDAT) treatment standards for selected hazardous wastes including listed refinery wastes. The treatment standards in the proposed ruling of April 8, 1988 are based on incineration treatment efficiencies derived from test burns of listed refinery wastes. Land treatment units must comply with the proposed rule unless they fulfill the requirements of a "no migration petition". Refineries without permitted land treatment units will be forced to arrange for incineration of their wastes prior to land disposal unless other technologies become commercially available which can meet the proposed BDAT standards. Due to limited incinerator capacity in the United States, the cost and difficulties in permitting incinerators, and recent emphasis on source reduction and recycling, alternatives to incineration which recycle the toxic constituents of the wastes are of interest to the petroleum industry.

The petroleum refining industry utilizes many different treatment/disposal schemes to manage listed hazardous wastes. The decision as to which scheme to use at a particular site, on a particular waste is a function of many factors, such as waste characteristics, available land, local environmental regulations, permitting, and public opinion. Clearly, there is no one treatment scheme which is applicable for all petroleum refining wastes, at all locations. Currently, the most widely used treatment technology within the industry for oily wastes is land treatment. However, Congress has included land treatment as a form of disposal under the land disposal bans. This study was undertaken in order to determine how effective other treatment technologies could be.

API has previously submitted comments (September 1986) to the EPA regarding technical problems associated with the Toxicity Characteristic Leaching Procedure (TCLP) and its application to oily wastes. The submission of TCLP data in this report should not be construed as a deviation from API's original position regarding TCLP. These data are submitted in an attempt to demonstrate the treatment efficiency of several technologies by applying the same TCLP criteria which were used by EPA in developing the solvents/dioxin rule (Federal Register November 6, 1986).

To provide a more comprehensive waste treatment study, technology vendors who were not API contractors were invited to submit data to the API for review and evaluation. In order to qualify for inclusion in the final report, these vendors had to treat listed refinery wastes, adhere to the API sampling and analytical protocols, and submit operating and test data by February 15, 1987. The only noncontracted vendor who met these criteria was one offering a pyrolysis treatment process. These data are included in this report.

API gratefully acknowledges the efforts of the Rocky Mountain Analytical Laboratory, which supplied all of the analytical data; the participating refineries, which provided listed wastes and treatment process data; the participating equipment vendors; Risk Science International, for writing the interim report; and members of the API Waste Technology Task Force, who planned the project and brought it to fruition. Special recognition is given to Hugh Dickey (Chevron) for the study design, Frank Prince, Ph.D., the API project manager, Piyush Shah, Ph.D. (Exxon) for structuring the analytical data tables, Richard Stalzer (Sohio) and Bill Deever, Ph.D. (Texaco) for the mechanical drawings, and Re'Naye Williams of API for typing the manuscript.

CHAPTER 1. EXECUTIVE SUMMARY

A number of technologies for the treatment of listed petroleum refinery oily wastes were evaluated in this study. The technologies studied were mechanical treatment (filtration), solvent extraction, thermal treatment (drying), chemical fixation and pyrolysis. In several cases the application of two treatment technologies in series was studied, e.g. filtration followed by drying, drying followed by fixation. No attempts were made to develop limits of operability for the subject treatment technologies nor was consideration given to the cost of various technologies. As evaluation tools, the reductions in mass from feed to product residues and the reductions in constituent leachate concentrations from feed to product residues were evaluated.

All of the technologies tested produced a residue of substantially reduced hazard, as measured by leachable concentrations (using TCLP) of hazardous constituents in the product solids. Four of the technologies also reduced the total constituent concentrations.

Table 1-1 allows a comparison of treatment efficiency among these technologies. It shows average residual concentrations (mg/kg) of ten indicator compounds in the product solids from four of the five treatment technologies which were evaluated. The order of decreasing efficiency, based on residual levels of organics, was: pyrolysis > solvent extraction > mechanical/thermal drying > mechanical treatment. The percent reduction for the eight organic compounds was 97-99%, for three of the four treatment technologies, based on average raw feed constituent data. Percent reduction values were lower for mechanical treatment. Metal levels were not changed significantly by any of the treatment processes. Actual percent reductions for each technology are shown in the summary tables of Chapter 8.

TABLE 1-1

AVERAGE CONCENTRATIONS OF TOTAL RESIDUE, mg/kg

	<u>Mechanical Treatment Total Residue Concentration</u>	<u>Solvent Extraction Total Residue Concentration</u>	<u>Mechanical Plus Thermal Total Residue Concentration</u>	<u>Pyrolysis Total Residue Concentration</u>
Benzene	46.5	1.3	0.55	ND
Toluene	304	5.0	3.0	0.01
Xylenes	568	4.4	2.4	ND
Anthracene	10.1	ND	53	ND
Chrysene	125	ND	62	ND
Naphthalene	144	0.005	46	ND
Phenanthrene	109	0.005	364	0.02
Phenols	4.1	ND	17	0.2
Chromium	654	1250	2450	44
Lead	128	260	275	6

ND : Not detected.



Table 1-2 allows a comparison of treatment efficiency among these technologies, based on the leachability of the product residues. It shows average concentrations (mg/L) of ten indicator compounds in the TCLP leachate from the product solids from the five evaluated treatment technologies. The order of decreasing efficiency based on the leachate concentrations of the eight organics was: pyrolysis > solvent extraction > mechanical/thermal drying > chemical fixation/thermal drying > chemical fixation > mechanical treatment. Drying the product solids prior to chemical fixation was more effective in reducing the leachability of the eight organic compounds. Chemical fixation was clearly the most effective in decreasing the leachability of the metals.

Figure 1-1 shows the percent average reduction in constituent weight and reduction in leachate concentrations of the raw feeds following treatment (mechanical, solvent extraction, mechanical plus thermal treatment, and chemical fixation or pyrolysis) for the three classes of compounds.

Results from the land treatment of refinery wastes are presented in another API report entitled, "Land Treatability of Appendix VIII Constituents Present in Petroleum Refinery Wastes" (API Pub. No. 4455). This study demonstrated the suitability of land treatment units to biodegrade organics and immobilize metals present in refinery wastes. Results of this land treatment study are compared to results from the treatment technologies in Chapter 8.

We believe that these two API reports provide technical support for the consideration of technologies other than incineration as best-demonstrated available technology (BDAT) for the treatment of refinery wastes.

TABLE 1-2

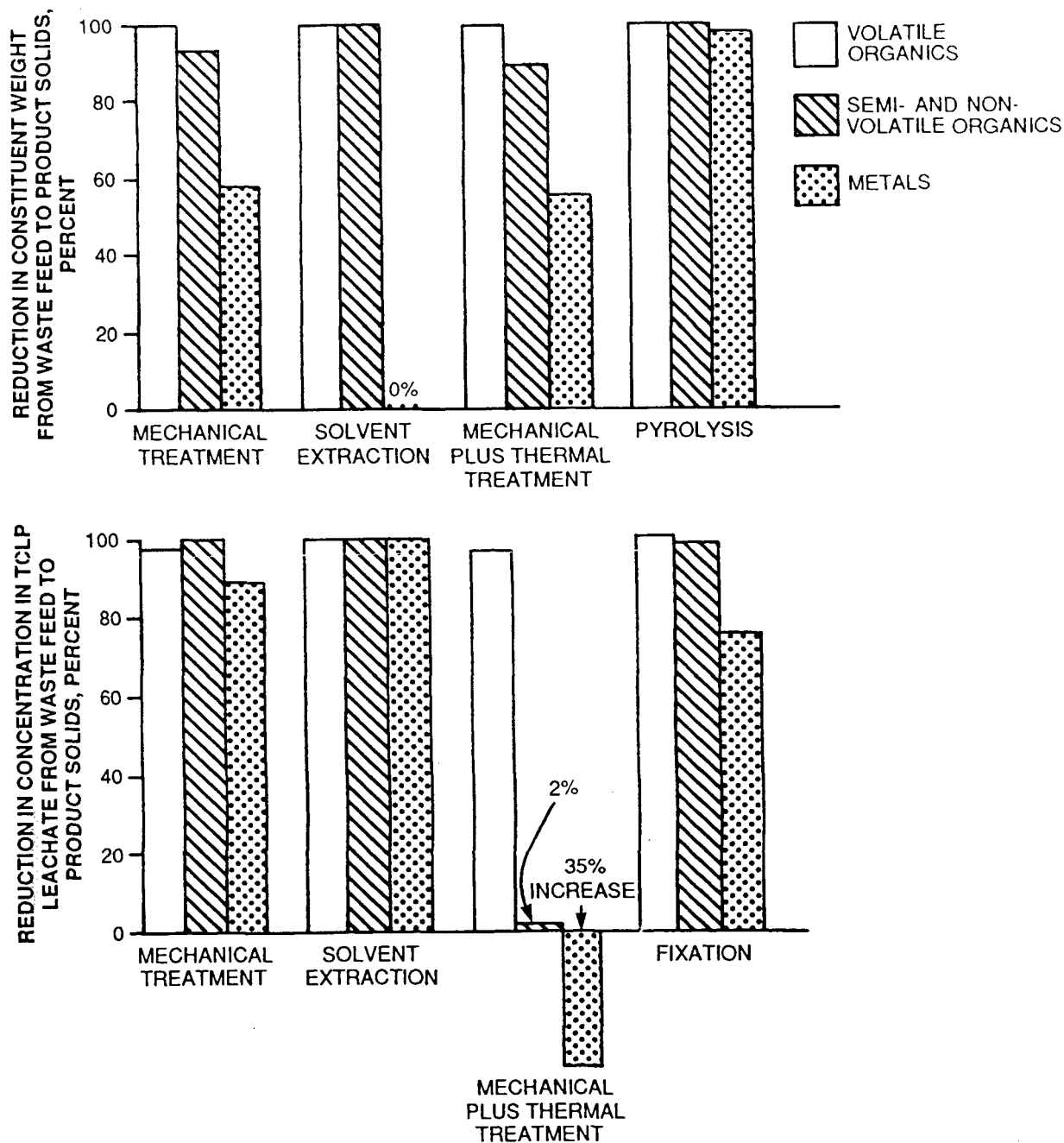
AVERAGE CONCENTRATIONS OF TCLP LEACHATE, mg/L

	Mechanical Treatment TCLP Concentrations	Solvent Extraction TCLP Concentration	Mechanical Plus Thermal TCLP Concentration	Fixation Product			Pyrolysis Residue TCLP Concentration
				Raw Feed TCLP	Cake Feed TCLP	Dried Cake TCLP	
Benzene	1.4	0.005	0.02	0.10	0.22	0.004	ND
Toluene	2.2	0.008	0.04	0.45	0.90	0.01	ND
Xylenes	1.5	0.01	0.07	0.60	0.82	0.02	ND
Anthracene	0.009	ND	0.01	ND	ND	ND	ND
Chrysene	0.024	ND	0.01	ND	ND	ND	ND
Naphthalene	0.17	0.007	0.06	0.19	0.10	ND	ND
Phenanthrene	0.009	ND	0.015	0.01	0.01	ND	0.004
Phenols	0.66	ND	0.27	0.53	ND	ND	ND
Chromium	0.045	0.11	0.05	ND	ND	ND	1.3
Lead	0.08	0.05	0.07	ND	ND	ND	ND

ND : Not detected.



FIGURE 1-1 REDUCTIONS IN CONSTITUENT WEIGHTS AND
TCLP LEACHATE CONCENTRATIONS



CHAPTER 2. SCOPE OF STUDY

This report presents the results of a study sponsored by the American Petroleum Institute (API) to evaluate the effectiveness of five technologies which were used to treat petroleum refinery wastes. Technologies specifically excluded from this study were incineration, which has been tested extensively by the U.S. Environmental Protection Agency (EPA), and land treatment, which has been studied and reported on separately by API (API Publ. 4455).

Feed and product samples were collected at various test sites where waste treatment was applied. These samples were subjected to physical/chemical analyses to determine the effectiveness of various treatment processes.

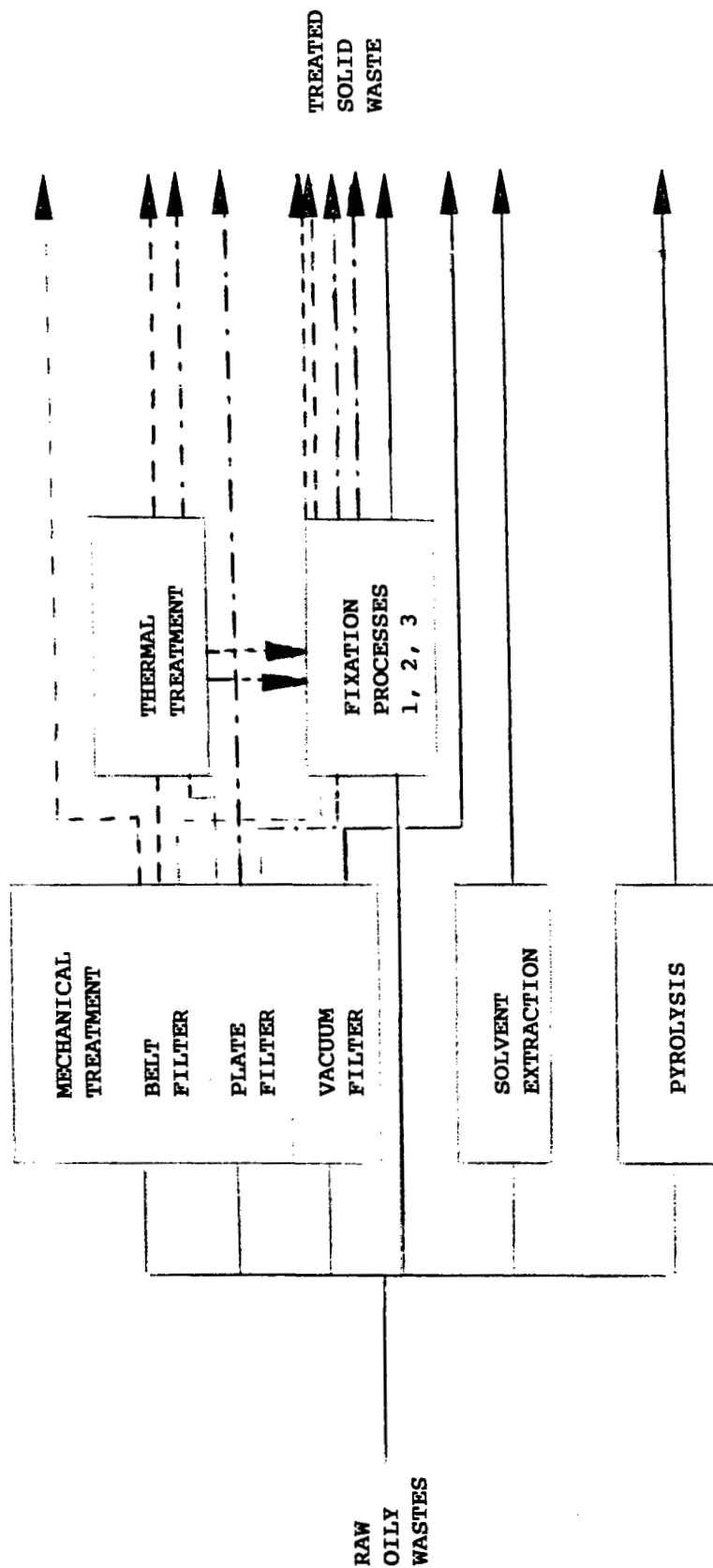
This chapter (2) of the report describes the scope of the study, the technologies, the analytical scheme and the methods of data correlation and interpretation. The approach regarding the test site selection and the scale of treatment operation (commercial, pilot or laboratory), the analyses performed and the quality assurance/quality control methods used are also described in this chapter.

Chapters 3 through 7, provide information on each of the five treatment technologies tested. Chapter 8 provides a comparison of the effectiveness of all of the technologies tested in this study.

A schematic of the study, the treatment processes and the sequences of processes tested, are shown in Figure 2-1.

FIGURE 2-1

SCHEMATIC OF WASTE TREATMENT TECHNOLOGIES EVALUATED



TECHNOLOGIES EVALUATED

Mechanical Treatment

Belt filters were tested at refineries C1 and C2, using listed oily wastes generated at those refineries. Operating conditions of each test were documented. Representative samples of waste feed, filtrates and filter cakes were transmitted to Rocky Mountain Analytical Laboratory (RMAL) for analysis according to EPA-prescribed protocols.

Plate filters were tested in a similar manner at refineries B1, B2, and B3. A rotary vacuum filter was tested at refinery V1.

Centrifuges are currently being used at a number of refineries but were not tested in this study for reasons of location plus the need to limit study costs.

Solvent Extraction

A solvent extraction process was tested which was thought to be representative of the general class of solvent extraction technologies. Tests were conducted on a 50-50 mixture of two listed wastes from refinery D. A batch pilot plant unit was used for this demonstration.

Thermal Treatment

A screw flight dryer was tested to represent this class of treatment technologies. Tests were conducted on belt filter cake from refinery C1, and on plate filter cake from refinery B1. Samples of each cake were treated at two temperatures--400°F (low temperature) and 650°F (high temperature). Tests were conducted in batch at a vendor's pilot-scale facility.

Pyrolysis

A rotary pyrolysis process was tested--in this case by the process vendor following protocols established by the API task force--to represent this class of treatment technologies. The tested feed was a mixture of three listed wastes from refinery E.

Fixation

Three different fixation processes were tested on samples at three different levels of pretreatment. Untreated oily wastes (API separator bottoms and slop oil emulsion solids from refinery A) were treated with fixation process 1. Belt filter cake from refinery C1 and plate filter cake from refinery B1 were treated using all three fixation processes: 1, 2 and 3. Thermally dried belt and plate filter cakes were treated using fixation process 2.

DATA AND CORRELATIONS

Figure 2-2 summarizes the types of analytical data obtained from the technology testing and the correlations of the data that were used to assist in interpreting the test results. The general approach to data analyses is discussed by category below.

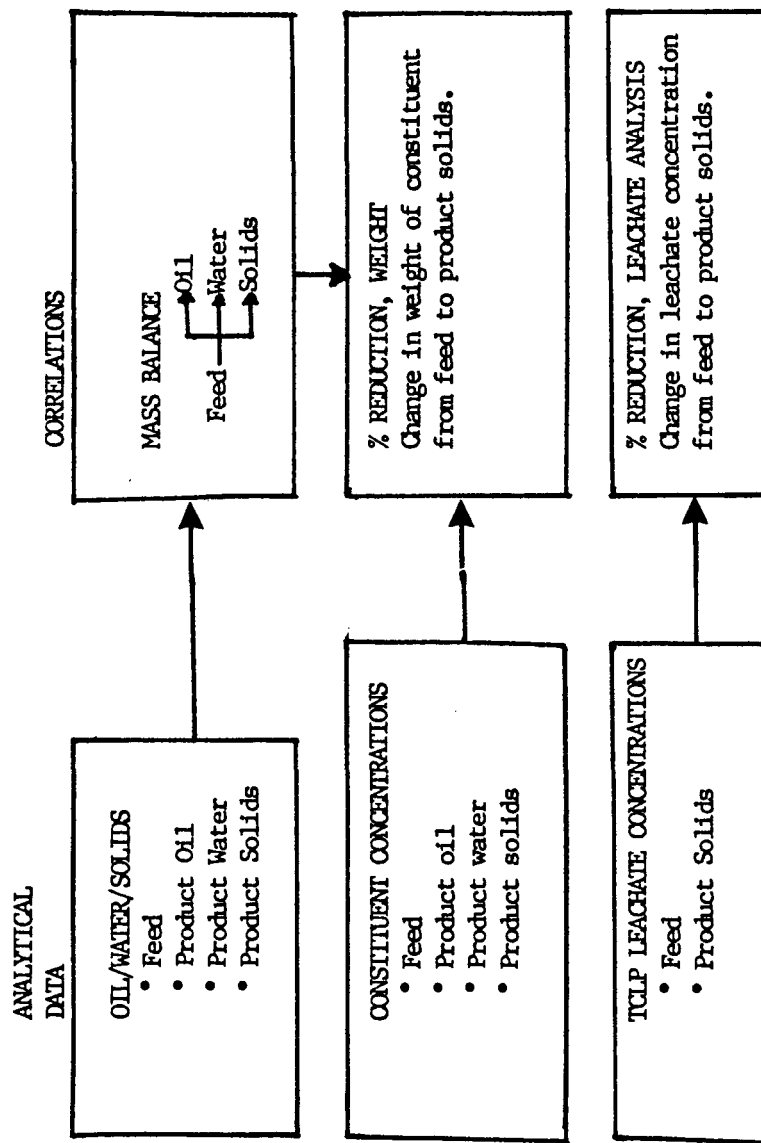
Analytical Data

As shown graphically in Figure 2-2, analytical data were of three types:

1. oil/water/solids analyses of the feed and products of each treatment technology;
2. analyses for Appendix VIII constituents in feed and products; and
3. Toxicity Characteristic Leaching Procedure (TCLP) analyses on feed and product solids.

FIGURE 2-2

ANALYTICAL DATA AND CORRELATIONS



More specific information on the analytical procedures used is given in a later section of this chapter.

Correlations

The analytical data listed above give a large measure of the effectiveness of the various treatment technologies: the concentration of Appendix VIII constituents in the product solids, and the TCLP analyses on product solids. Two additional measures of treatment efficiency were developed: reduction of constituents by weight, and percent reduction in leachable concentrations. The first of these, Percent Reduction (Weight) is derived from a combination of the mass balance (for the most part calculated from oil/water solids analyses) and the constituent concentrations in feed and product solids. When combined, these data allowed the reduction in weight of constituents from feed to product solids to be calculated.

Each process was evaluated using this mass balance approach to determine the bulk amount of hydrocarbons or toxic constituents physically removed from the waste and recycled. The main objective was to generate data which would provide a relative scale of the removal efficiencies of the generic processes, whether or not they could be considered viable BDAT technologies.

The wastes varied substantially in composition from very low to very high oil content. This was desirable because it gave a good range of wastes that would be expected to be processed through these units. However, this variation discourages absolute comparisons within a generic technology (e.g. plate versus belt filters) because some wastes were inherently easier to separate than others.

The second measure of treatment efficiency, Percent Reduction (Leachate Analysis), is derived from TCLP analyses on feed and

product solids. The reduction in leachable concentrations of constituents from the feed to the product solids was determined.

LOCATION/SCALE/TECHNOLOGY SELECTION

A goal of this study was to test commercial-scale technologies, where possible, to demonstrate industrial application, availability and treatment performance. All tests on mechanical treatment equipment were conducted on commercial equipment operating at petroleum refineries. The solvent extraction process was tested on a pilot unit using a mixture of refinery sludges. A possible alternative choice would have been to test the commercial scale unit which was operating at a Superfund site. The decision was made that the testing on refinery wastes on a pilot scale was preferable to testing non-refinery wastes on a commercial scale since the two wastes had only a few constituents in common.

Thermal treatment was tested using refinery wastes on a pilot unit from an equipment vendor. Industrial operations other than petroleum use thermal driers on a commercial scale but no such operation exists treating refinery sludges. Feeds which were thermally treated were filter cakes. These were product solids derived from filtration of raw refinery wastes. Use of filter cake was based on a most probable scenario, but does not indicate any technical reason against feed of raw wastes to this equipment.

Pyrolysis and fixation tests were conducted at the laboratories of the process vendors. The process vendors were confident of their ability to scale up the tests based on experience with other feedstocks. For fixation, no advantage was seen in larger scale tests--all were at lab scale.

Selection had to be made among a wide array of process and equipment vendors based on a limited budget and time schedule. Results of the five technologies which were evaluated by API are

likely to be indicative of generic classes or groups of processes and equipment.

ANALYTICAL PROCEDURES

The following sections give more information on the analytical tests and techniques and also summarize the quality assurance/quality control methods of the study.

Analytical Techniques

The oil/water/solids analysis employed a method developed by Chevron Corporation (Modified Oven Drying Technique or MOD-T.). The method is based on a low temperature distillation of the sample to generate a volatile oil and water fraction which is subsequently condensed. The remaining material is extracted with methylene chloride to generate a nonvolatile fraction, with solids defined as the solvent insoluble residue. This was the method of choice over EPA methods (i.e. 418.1, 3540 or 3550) specifically because of the potential for loss of volatile hydrocarbons by the EPA procedures. Consequently, results may differ among the test procedures, with the MOD-T reflecting somewhat higher oil recovery levels. A copy of this procedure is in the appendix.

Table 2-1 shows Appendix VIII constituents of refinery wastes which were analyzed in feeds, products and TCLP leachates. Analytical methods were derived from three sources of EPA methods:

- 1) the methods promulgated in 40 CFR 136 for priority pollutants;
- 2) the methods published in SW-846; and
- 3) methods published by EPA for Superfund investigations.

A subset of the Table 2-1 lists is shown in Table 2-2. This list was developed to allow a screening test as an economy measure. These compounds termed "indicator" or "screening" compounds were measured by alternative techniques.

TABLE 2-1

APPENDIX VIII CONSTITUENTS ANALYZED

Volatile Organics

Benzene
Carbon disulfide
Chlorobenzene
Chloroform
1,2-Dibromoethane
1,2-Dichloroethane
1,4-Dioxane
Methyl ethyl ketone
Styrene
Ethylbenzene
Toluene
Xylene, m
Xylenes, o & p

Acid Organics

Benzenethiol
o-Cresol
p & m-Cresol
2,4-Dimethylphenol
2,4-Dinitrophenol
Phenol

Metals

Antimony
Arsenic
Barium
Beryllium
Cadmium
Chromium
Cobalt
Lead
Mercury
Nickel
Selenium
Silver

Base/Neutral Organics

Anthracene
Benzo(a)anthracene
Benzo(b)fluoranthene
Benzo(j)fluoranthene
Benzo(k)fluoranthene
Benzo(a)pyrene
Bis(2-ethylhexyl)phthalate
Butyl benzyl phthalate
Chrysene
Dibenz(a,h)acridine
Dibenz(a,h)anthracene
Di-n-butyl phthalate
o-Dichlorobenzene
m-Dichlorobenzene
p-Dichlorobenzene
Diethyl phthalate
7,12-Dimethylbenz(a)anthracene
Dimethyl phthalate
Di-n-octyl phthalate
Fluoranthene
Indene
Methyl chrysene
1-Methylnaphthalene
Naphthalene
Phenanthrene
Pyrene
Pyridine
Quinoline

TABLE 2-2

CONSTITUENTS ANALYZED AS INDICATOR OR SCREENING COMPOUNDS

Volatile Aromatics

Benzene
Ethylbenzene
Toluene
Xylenes*

PNAs/Phenols

Anthracene
Chrysene
Naphthalene
Phenanthrene
Phenols*

Metals

Chromium
Lead

* Indicator TCLP measures total xylenes and total phenols (sum of phenol, cresols, and 2,4-dimethylphenol).

TCLP leachates were prepared using the method in Appendix I to 40 CFR Part 264. Simply stated, the TCLP procedure is designed to generate an aqueous leachate of a waste. Leachates were prepared at a 20 to 1 ratio relative to the solid material in the sample. The leachate was then analyzed for the various target parameters. Results are reported in mg/L in the leachate.

The initial leaching procedure requires two separate laboratory preparations (extractions), one for volatile organics and one for the remaining parameters. The preparation for volatile organics requires the use of a specially designed device, termed the zero headspace extractor (ZHE).

The initial step in performing a TCLP extraction is the pressure filtration (50 psi) of the sample through a 0.8 micron filter. The solid phase remaining after this filtration is then mixed with the aqueous TCLP extraction fluid using a 20 to 1 ratio. After 18 hours of "extraction" the solid/leachate mixture is filtered a second time. The filtered leachate from this step is then combined with any filtrate from the initial filtration.

For wastes containing "oil," the initial filtration often results in a two-phase filtrate containing oil and water. According to the TCLP protocol, the oil phase must be analyzed separately, and the results mathematically combined with those from the extract. Analyses of the various leachate solutions were performed according to EPA procedures described in Appendix I (40 CFR Part 264).

Samples of fixed waste materials were ground to pass a 5.55 mm (0.375-inch) standard sieve prior to conducting either the Total TCLP or the Indicator TCLP test as required by the EPA protocol (51 FR 40643).

Quality Assurance/Quality Control

All laboratory analyses were performed according to specifications in a Quality Assurance Project Plan (QAPP), as specified in EPAAR 1552.246-71. The QA/QC plan of the laboratory (RMAL) for this project followed the elements of their generic laboratory-wide quality insurance procedures for sample preparation and analyses. A separate QC Data Summary Report has been prepared by RMAL, which presented the QC results that were directly related to the performance of the methods on these samples. Other QC activities such as calibration, mass tuning checks and activities related to the general performance of the instruments have not been reported, but are archived in the report files at RMAL.

Quality control analyses consist of the following activities which are included in the QC Data Summary Report:

- multipoint standard calibration;
- analysis of blanks;
- analysis of spiked and duplicate samples;
- analysis of standard reference materials;
- daily calibration, including mass spectrometer tuning checks (BFB and DFTPP), where appropriate; and
- addition of surrogate spikes into each sample for GC/MS analyses.

Sufficient amounts of representative samples were sent to the laboratory with the following history:

- All samples were collected in glass sample bottles;
- No chemical preservation was used, and the samples were stored at 4°C until analyzed;

- Samples for volatile organics were collected with minimal headspace; and
- Analysis was performed in an expeditious fashion, applying 40 CFR 136 water holding times where appropriate.

A chain of custody record was established for each sample except as noted in the QA/QC report.

CHAPTER 3. MECHANICAL TREATMENT

INTRODUCTION

Petroleum refinery wastes are generally oil-in-water emulsions stabilized by the presence of finely divided solids. By removing the solids, the emulsion is often broken, allowing recovery of two distinct liquid phases, an oil-rich phase and a water-rich phase, as well as a concentrated-in-solids cake. Mechanical treatment is a well-demonstrated method to accomplish this phase separation.

The toxic components of the waste, organic compounds and metals (Table 3-1), will partition into these three phases according to whether they are solid or liquid; their solubility in water and organics; and the efficiency of the separation device in separating solids and liquids.

The mechanical treatment methods evaluated in this study were all filtration methods. Filtration is the process of separating suspended solid material from a liquid by forcing the liquid through the voids of a porous mass called the filtering medium. Two important variables in filtration are the material that forms the separating medium and the method used for forcing the liquid through this medium. The filtration methods used in this study--the belt filter, the recessed plate filter and the rotary vacuum filter--demonstrate a wide range of these two variables.

In the belt-filter, the filtering medium can range from a screen to a cloth, and driving force for liquid movement is first gravity and then mechanical pressure of a screen or cloth pressing against the solids. In a recessed plate filter, the medium is a cloth, sometimes with a porous granular solid or precoat deposited on the cloth, and the driving force is the discharge pressure of a pump. In a rotary vacuum filter, the medium is usually a pre-

TABLE 3-1

CONSTITUENTS OF REFINERY WASTES

Volatile Organics

Benzene
 1,4-Dioxane
 Methyl ethyl ketone
 Styrene
 Ethylbenzene
 Toluene
 m-Xylene
 o- & p- Xylenes

Acid Organics

o-Cresol
 p- & m-Cresol
 2,4-Dimethylphenol
 Phenol

Metals

Arsenic
 Barium
 Cadmium
 Chromium
 Lead
 Mercury
 Selenium
 Silver

Base/Neutral Organics

Anthracene
 Benzo(a)anthracene
 Benzo(b)fluoranthene
 Benzo(a)pyrene
 Bis(2-ethylhexyl)phthalate
 Chrysene
 Dibenz(a,h)anthracene
 Fluoranthene
 Indene
 1-Methylnaphthalene
 Naphthalene
 Phenanthrene
 Pyrene

coated cloth while the driving force is provided by drawing a vacuum on the product liquid side of the cloth.

Depending on the feed waste characteristics and the filtration process chosen, a filter cake results which may vary from 20 to 60 percent solids. A product filtrate stream is produced also which, in all the study cases, separated into an oil phase and a water phase.

OVERVIEW

The data gathered in this program demonstrate the potential effectiveness of mechanical treatment of wastes at petroleum refineries for both economic and environmental reasons.

From an economic standpoint, the most important factors are recovery of oil, which would otherwise be lost, and reduction of the quantity of waste to be disposed.

Oil recovery from the oily sludges tested ranged from 80 to 99%. To put this in perspective, if a refinery produces 50 tons per day of oily sludge with an oil content of 10%, mechanical treatment would recover about 10,000 barrels (42 gal/barrel) of oil per year.

The mass reduction accomplished by mechanical treatment ranged from 85-96%. The actual reduction varies almost inversely with the solids content of the feed waste. Again solely for illustration: if a refinery produces 50 tons per day of waste and can reduce the weight by 90%, only 5 tons of cake is disposed.

The environmental benefits of mechanical treatment are reflected in the mass reduction of Appendix VIII constituents between feed and cake and the reduced concentration of these same Appendix VIII components in the TCLP leachates of feed and cake. For the organic components, both of these reductions--mass balance reduction and TCLP leachate concentration reduction--range from 90 to more than 99%, with only a small number of exceptions. For the volatile organics, average reductions in both mass and leachate analysis are from 97 to 99%. For the metals, the reductions are not so great, but perhaps this is not as significant, since metals can be "fixed" chemically in an added treatment step, or can be immobilized by cation exchange on soil.

Overall, mechanical treatment of refinery wastes yields a much reduced weight of waste which is less toxic and less mobile than the original waste.

Integration of mechanical treatment into an operating refinery would be quite straight forward. Product water can be included readily into the refinery's wastewater treatment system. Product oil can be recycled into the oil processing operation.

DISCUSSION--EQUIPMENT DESCRIPTION AND OPERATION

In this section the three different types of filters are described and the principles of operation and important operating parameters for each are reviewed.

Belt Filter

Applicability: Belt filters have been widely used for many years in the dewatering of sludges from the pulp and paper industry and from municipal sewage treatment plants. Usage for other industrial sludges has increased dramatically over the past 10 years because of the large reduction in volume of residues that is accomplished. Many petroleum refineries have found that belt filter press treatment provides the added benefit of oil recovery from the separated liquid phase.

The product streams generated from the belt filter press are filter cake and filtrate, and the filtrate may separate into oil and water phases.

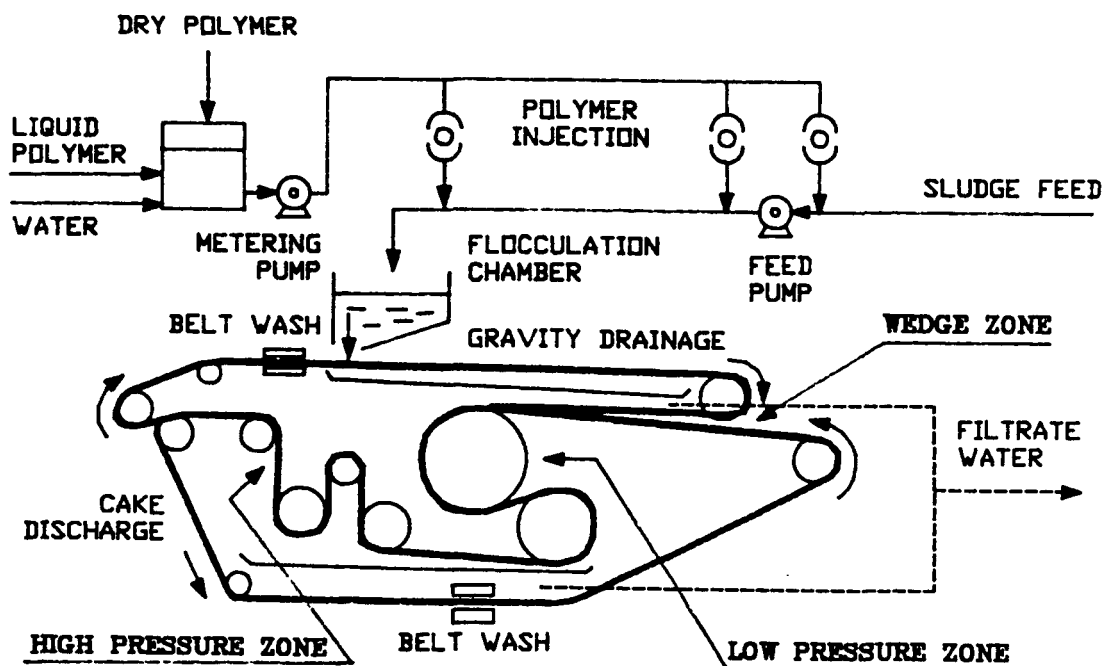
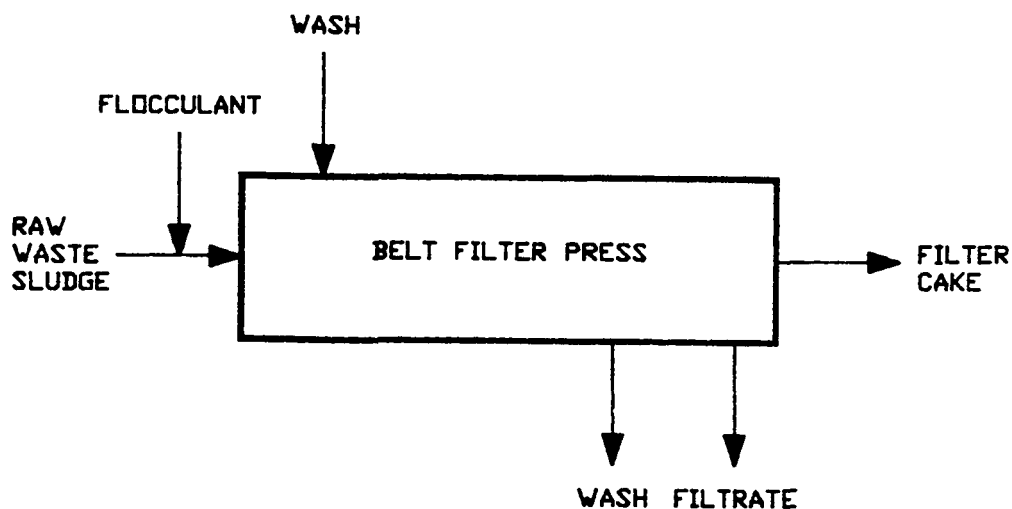
Underlying principles of operation: Belt filtration processes include three basic operational stages: chemical conditioning of the feed slurry, gravity drainage to a nonfluid consistency, and compaction of the predrained sludge. A schematic diagram of the

belt filter process is shown in Figure 3-1. After addition of a polymer (polyelectrolyte) and subsequent flocculation, the feed material is applied to a solids retaining membrane or filter cloth. As the feed moves through the filter, liquid flows into filtrate collection pans, and solids are retained on the filter cloth. A cake is produced for disposal.

Initially, the raw feed is mixed with a polymer to cause flocculation of solids and enhance separation of the liquid phase. The preconditioned feed is then distributed to a porous, woven belt where the gravity phase of liquids/solids separation occurs. Free liquid flows through the porous belt by gravity and into filtrate collection pans. Up to 60% of the liquid phase of the feed can be removed in the "gravity zone." The porous belt with the gravity-separated solids on it is then enveloped by another belt as it leaves the gravity zone and enters the low pressure or "wedge zone." Further phase separation is achieved here using mechanical pressure. In the third zone, the belts pass through a series of large rollers that squeeze the belts together, forcing additional liquid through the porous belt into filtrate collection pans. Manufacturers report that pressures can reach 14.2 pounds per square inch or more in the high pressure zone as the belts travel through a series of rollers. The final liquids separation occurs here due to the high pressure and shear forces between the two belts as they travel over the rollers. Pressures in the second and third zones are generally adjustable. The deliquified sludge is scraped off the belt into a container for disposal. A high pressure water wash is usually applied to the belt at this point to clean it and to unplug clogged belt pores.

The polymer used to flocculate solid materials in the feed is mixed with water and can be injected into the feed at several injection points. Often a cationic polymer is used. Oil refiners who have experience in deliquifying oily wastes are able to produce a solids cake with 20-40% oil, depending on feed characteristics.

FIGURE 3-1
DIAGRAM OF BELT FILTER PRESS PROCESS



Operating parameters affecting performance: Adjustment of the process variables for "optimum" operation of a belt filter within a refinery is dependent on feed characteristics which change often, sometimes constantly. Within a modern, complex refinery, feed characteristics (e.g. oil and solids content, flocculation characteristics) are always changing and re-optimization of operating parameters must be done frequently.

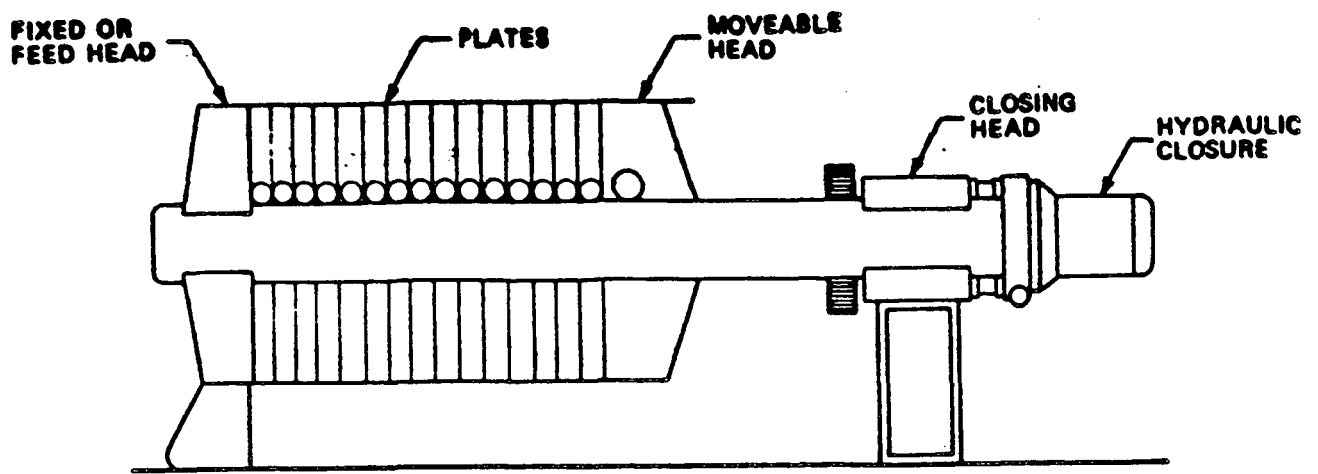
The important process variables for proper operation of the belt filter are as follows:

- waste composition and characteristics
- flocculation efficiency depending on choice and rate of polymer, point and energy of mixing
- belt material, pore size, weave
- feed rate and belt speed (these are interdependent.)
- pressure exerted on the sludge

Plate Filter

Applicability: The recessed plate filter (a modification of the plate and frame filter) has been used for many years in both product processing and sludge treatment. Industries that have used plate filters include petroleum refining, chemical, pharmaceutical, metal finishing, aviation, steel manufacturing, transportation (oily sludges from railroad and motor oil), food (used oils), and paint. Plate filters are very effective at breaking emulsions, if the feed is properly conditioned.

Underlying principles of operation: A plate filter press is a batch filtration process used to separate solids or particulate material from a liquid stream. Figure 3-2 is a schematic process diagram. The recessed plates are connected to form a series of chambers. The filter medium is supported on the plates; the recessed chambers provide a cavity for the collection of solids filtered out of the influent.



SCHEMATIC SIDE VIEW OF A RECESSED PLATE PRESSURE FILTER

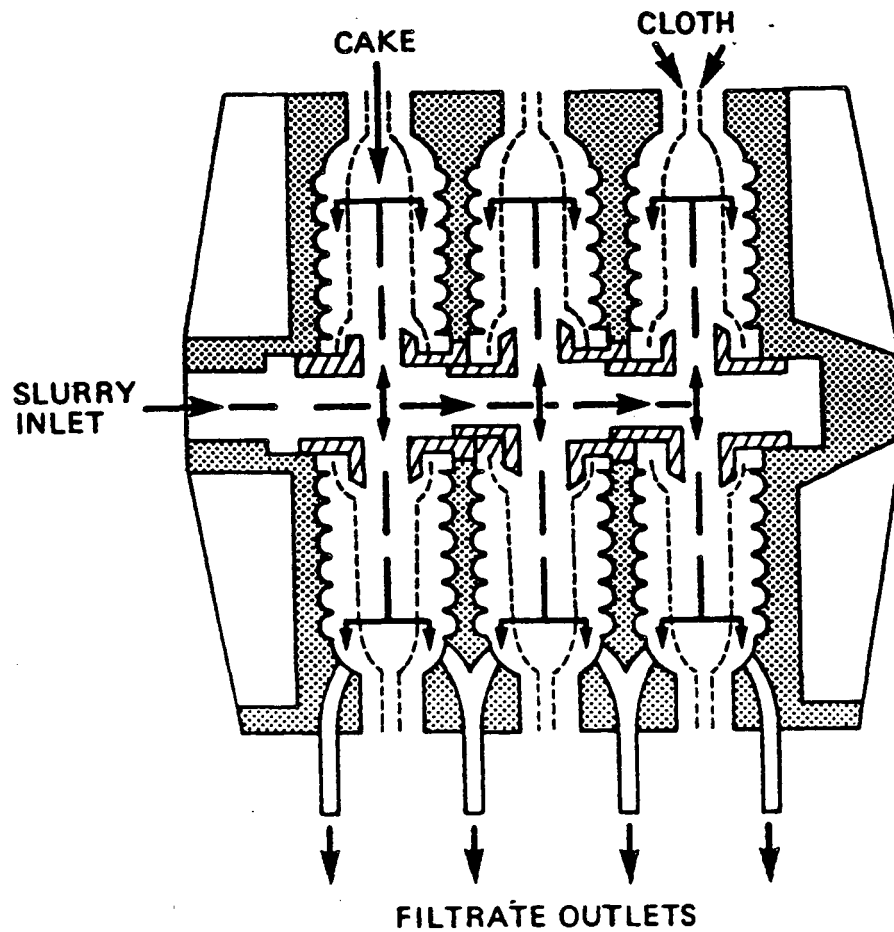


FIGURE 3-2
CROSS SECTION OF A FIXED-VOLUME
RECESSED PLATE FILTER ASSEMBLY

Feed sludge is normally conditioned externally by addition of lime and sometimes iron salts (e.g. ferric chloride). The conditioned feed enters through a feed port, typically in the center of the plate, and is fed in parallel to each filter chamber. Feed pressures are 100-250 psig. The plates are pushed together by a piston on the moveable head and held together at 2,000-10,000 psig. The liquids pass through the filter media and are collected through outlet ports on each plate. The most frequently used filter media for refinery sludges are filter cloths of woven synthetic materials, such as nylon or polypropylene. Quite often the filters are precoated with diatomaceous earth to reduce cloth blinding, enhance solids/liquids separation and emulsion breaking and to ensure quick cake release when the filter is opened. The cake collects in the chambers formed by the two plates coming together. When the chambers are full, a wash stream can be injected. The cake is removed by moving the plates apart and dislodging the cake into a container. Any cake remaining on the plate may be scraped off. Air can be introduced behind the media cloth on both sides of each plate to assist in cake removal. Other variations to ease operations are also available.

Operating parameters affecting performance: The important process variables for proper operation of a plate filter press include:

- waste composition and characteristics
- pressure (usually up to 100 to 250 psig)
- type of filter cloth (usually nylon or polypropylene) and weave
- media precoat (usually diatomaceous earth) and thickness
- cycle time (fill time, filtration time, and cake release time)
- influent conditioning (e.g., dosage of lime and/or iron salts)

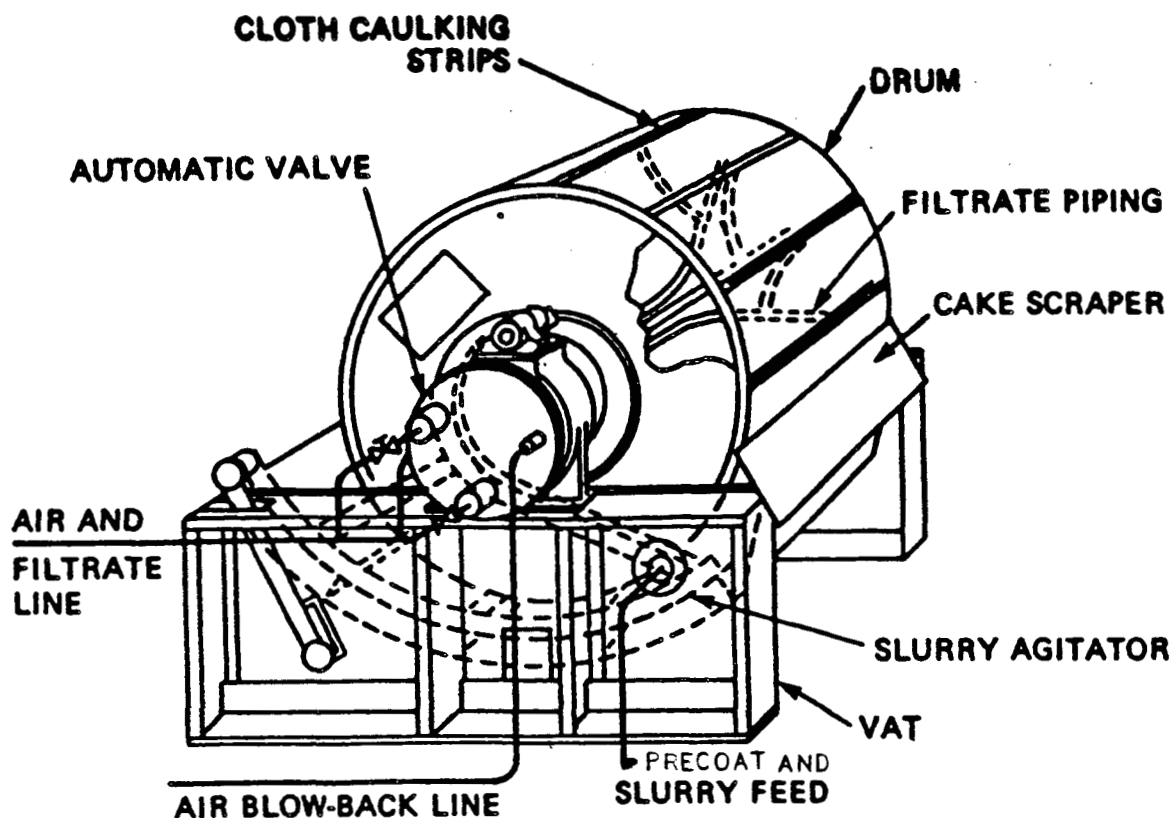
- influent temperature
- cake thickness
- initial flow rate (gal/hr-ft²) and solids loading (lb/hr-ft²)

Rotary Drum Vacuum Filter

Applicability: The rotary drum vacuum filter has been used in industry for many years in both product processing and dewatering of sludges. Industries that have used rotary drum vacuum filters include sugar refining, metallurgical industry, petroleum refining, and municipal sewage treatment plants. Product streams generated from the vacuum filter are filter cake and filtrate.

Principles of operation: The rotary drum filter (Figure 3-3) consists essentially of a cylindrical drum supported by an open-tank system. The axial rod of the rotary drum is placed on the open tank to allow rotation of the drum. The placement of the drum is such that its lower portion is confined within the tank walls, while the upper portion is exposed to the atmosphere. The drum shell is composed of a number of shallow compartments covered with a drainage grid and a filter screen which is held in place by lateral caulking. The interior of the compartment is connected to a valve mechanism which, during operation, automatically applies either negative (vacuum) or positive pressure to the several conduits in rotation. The automatic valve is connected to a vacuum system and to a source of compressed air.

A slurry agitator is suspended at the bottom of the tank to mix the feed. The filter cake is discharged from the drum surface by a scraper blade containing a detachable rubber tip. Normally a precoat is applied to the drum before sludge feed is started. Some filters are enclosed to reduce heat loss and control air emissions.



**CUTAWAY VIEW OF A DRUM OR SCRAPER-TYPE
ROTARY VACUUM FILTER**

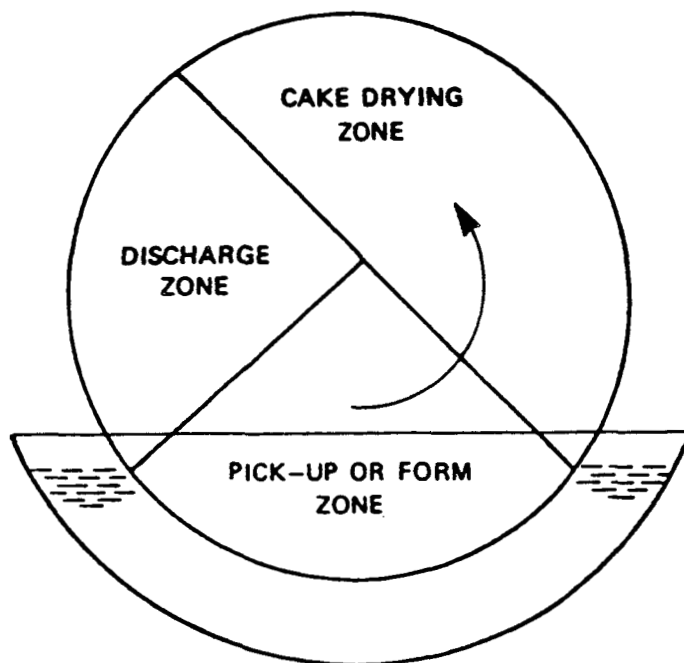


**FIGURE 3-3
OPERATING ZONES OF A ROTARY VACUUM FILTER**

The drum rotates slowly while the tank is constantly fed with the sludge. The level in the lower tank is maintained at a constant depth. The valve is set for the timing required in each operation. Through the action of the valve, vacuum is applied to those compartments of the drum passing through the sludge. The vacuum created within the compartments causes a flow of filtrate through the filtering medium. This will allow a layer of solids (cake) to deposit upon the filter screen. As the drum rotates, the vacuum in the compartments is maintained. Finally, the cake is scraped from the precoated filter medium. The scraper is slowly advanced into the precoat layer so that a fresh face of unblinded precoat is exposed each rotation. The filtering cycle is complete when most of the precoat has been removed and a new deposit of precoat is required.

Operating parameters affecting performance: The important operating parameters for the vacuum filter press are as follows:

- Sludge composition and characteristics
- Rotation cycle of the drum in ft/min
- Media precoat and thickness
- Influent conditioning
- Temperature of the feed
- Cake thickness
- Hydraulic and solids loading
- Viscosity of the filtrate
- Precoat layer removal rate (knife advance or cut rate)

TEST PROCEDURES

Belt Filter

Two belt filters currently in use at two operating refineries were evaluated in this study. The processes differ slightly as described below.

Refinery C2 At refinery C2, API separator sludges (K051) were treated with a belt filter in two separate tests. The equipment has separate belts for the gravity and pressure filtration sections. The gravity section is 84 inches wide and 11.1 feet long, with an effective area of 74 square feet. The pressure section consists of 14 rollers, the first 36 inches in diameter, the second 18 inches in diameter and the others 12 inches in diameter. The effective area is 39 square feet, and the overall dimensions of the pressure section are 44 inches wide by 80 feet long.

Test sludges were collected from API separators using vacuum trucks, and transferred to a 500 bbl portable tank. Operating parameters for the tests were as follows:

Polymer (cationic)	1.3% solution; mixed with sludge prior to gravity section of press
Total feed rate	26 gpm (1.5 gpm polymer solution, 3 gpm dilution water, 21.5 gpm sludge)
Polymer concentration in feed	750 ppm
Belts	63.5 x 30.5 mesh 0.0748 open area
Belt tension	200 psi (10 lb/linear inch)
Belt speed	20 ft/min in gravity section 35 ft/min in pressure section

The gravity and pressure filtrates were collected in separate portable tanks prior to backwashing the belts.

Refinery C1: At refinery C1, operating parameters for the belt filter test were as follows:

Sludge feed rate	61 gpm (0930 hr); 75 gpm (1300 hr)
Washwater	100 gpm, 96 psig (constant)
Feed Temperature	85°F

Polymer	1.5% solution
	230 gph (0930 hr); 225 (1300 hr)
Belt tension	
Top belt	11 psig
Bottom belt	12 psig
Belt speed	12 ft./min.

The operating conditions shown for the two belt filter presses tested are within the ranges typically used at refineries for these types of oily waste feeds.

Plate Filter

Three plate filters currently in use at three operating refineries were evaluated in this study. The processes differ slightly as described below.

Refinery B2: At refinery B2, a mixture of listed refinery wastes was treated in a plate filter. The waste feed was drawn directly into the press from a holding tank. The tank typically contains a mixture of dissolved air flotation (DAF) sludge (K048), API separator sludge (K051), slop oil emulsion solids (K049), and miscellaneous oily materials such as tank bottoms. This mixture was conditioned with lime, at a dosage of about 2.5% of total sludge feed. For the tests reported here, the filter cloth in the press was a satin weave nylon. No precoat was used.

The filtration cycle began with 12 minutes of "fast fill" at 565 gpm, after which the flow rate was halved. Total filtration time was 3 hours and 45 minutes at a final pressure of about 210 psig. This was followed by a 23-minute hot water wash and 20 minutes to allow lines to drain before the filter chambers were opened. Release of the filter cakes took an additional 20 minutes. Throughout the filtration cycle, the temperature was approximately 145°F. At the end of the hot water wash, the filtrate temperature reached about 195°F.

Feed samples were drawn from the holding tank before the run and about 30 minutes, 2 hours and 3 hours after the run had started. At the end of the cycle, cakes were released to a conveyor system which transported the entire cake volume to a clean dumpster. This cake volume was core sampled in 20 locations on a 4 x 5 point grid. These samples were composited in a clean plastic-lined bucket from which sample containers were filled. The sampling procedure was completed within 20 minutes after all cakes had been released.

Refinery B1: At refinery B1, a mixture of about 99% API separator sludge (K051) and 1% tank bottoms (leaded and unleaded) were tested in a plate filter. The filter consists of 99 epoxy-coated recessed chamber plates. A synthetic fabric is used as the filter medium and typically, the sludge is pressurized to 225 psi by hydraulic force. The filter cakes fall into a dumpster located directly below the filter.

Feed solids are initially reduced in size with a comminutor, after which the feed is pumped to decant tanks for solids settling and sludge thickening. The thickened sludge is then pumped to the plate filter press for solids/liquids separation. A 50/50 mixture of silica and alumina is used as a precoat. The feed is conditioned by addition of lime to obtain about 15% solids in the feed and to enhance filterability of the cake. Plant experience on this waste has shown that 15% solids is optimal; a lower solids content would require a longer cycle time.

The precoat is applied to the filter medium by first filling the filter with water. Precoat slurry is then pumped into the filter, and the filtrate is released allowing the precoat to coat the filter medium to about 1/16" thickness. The lime conditioned sludge is then pumped into the filter from both ends, forcing the

liquid in the precoat slurry out of the filter. The dual feed entry prevents the plates from warping and more evenly distributes the sludge on the press. When the filtrate flow falls below 32 gpm, the filter cycle is complete and the filter is ready to be opened. The accumulated cake falls from the opened plates through a hole in the floor to an awaiting truck or dumpster. The filter is then backwashed with water; when necessary the filter medium is washed with a high-pressure spray gun.

A typical cycle time for the filter is 3 to 4 hours, depending on the feed characteristics. On an average day, about 28,000 gallons of sludge are processed, generating about 40 tons of filter cake. The average solids content in the cake is about 55 percent.

Refinery B3: At refinery B3 mixed sludges are pumped to a mixed conditioning tank. Lime plus a proprietary agent are added at a rate of 0.7-1.0 pounds per pound of feed solids.

The plate filter has a capacity of 96 cubic feet in 100 chambers, 1" thick, formed by polypropylene plates. The filter is precoated with diatomaceous earth to a thickness of 1/16 inch.

Filter feed rate starts at 135 gpm with rate decreasing over a cycle of 90 to 120 minutes until a filter inlet pressure of 225 psig is reached.

Rotary Drum Vacuum Filter

One vacuum filter, currently in use at an operating refinery, was evaluated in this study.

Refinery V1: At refinery V1 slop oil emulsion sludges (K-049) were treated with a vacuum filter in two separate tests. The rotary drum is 8-feet in diameter and 12-feet long. It is

operated by a 40 HP vacuum pump which has a capacity of 850 SCFM. The vacuum rotary drum has a speed of 2-12 minutes/revolution and is driven by a 3 HP drive. The vacuum filter has an adjustable knife to provide effective scraping of filter cake.

Test sludges were collected from the filter and samples were taken for analysis. Operating parameters for the test were as follows:

Feed Temperature	= 130°F
Feed Rate	= 6 gal/min
Drum speed	= 4.3 min./rev.
Conditioning of feed	None
Knife advance	= 0.0005 inches/sec
Precoat about 2 inches thick	when sampled
Vacuum level	= 17-18 in. Hg
Feed tank	= 2,000 bbl, with two mixers

TEST RESULTS AND DISCUSSION

The types of data obtained from the mechanical treatment tests include:

- Percentage of oil, water and solids in the waste feed
- Percentage of oil, water and solids in each of the three product streams (i.e., "oil", "water", and filter cake)
- Concentrations of specific volatile organics, base neutral organics, phenols and cresols, and metals in the waste feed and product streams
- Concentrations of specific volatile organics, base neutral organics, phenols and cresols, and metals in the TCLP leachate derived from the waste feed and product filter cakes

For each of the filters tested, measurements were made of a subset of the data listed above. The results for each individual test are presented.

The results have been presented in several ways to allow evaluation of how effectively the various treatment processes reduce environmental hazards. Three methods of evaluation have been used, where sufficient data are available.

1. Concentration of selected constituents in the waste feed and the filter cake have been presented.
2. Using the concentration data discussed in 1., and a mass balance defining weight of filter cake as a fraction of weight of raw feed, "percent reduction, weight" has been calculated and presented. The "percent reduction, weight" shows what percentage of a constituent present in the feed has been removed, and is not present in the filter cake.
3. The concentrations of specific constituents in the TCLP leachate from the waste feed and the filter cake is given and the "percent reduction, leachate analysis" has been calculated.

In order to arrive at the mass balance required to complete 2. above, development of a calculated material balance was required. Since all of the mechanical treatment work was done in continuous operation, on industrial scale equipment and within the refineries, mass balance by weight was not possible; the equipment required was not in place. The balances were calculated from the oil/solids/water analyses on filter feed and filter products. It should be appreciated that these are calculated balances and not measured balances.

As a first step toward the material balances, the oil/water/solids analyses were normalized to add up to 100%.