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**DEVELOPMENT OF CLEAN COAL AND CLEAN SOIL TECHNOLOGIES USING
ADVANCED AGGLOMERATION TECHNOLOGIES**
Volume 3—Soil Clean-up and Hydrocarbon Waste Treatment Process

By
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January 1990

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Pittsburgh, Pennsylvania

By
Alberta Research Council
Devon, Alberta, Canada

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ARC/EPRI Consortium

Coal Processing Development Program

**Development of Clean Coal and
Clean Soil Technologies Using
Advanced Agglomeration
Techniques**

**Volume 3 – Soil Clean-up and
Hydrocarbon Waste Treatment
Process**

DE-FG22-87PC79865

Prepared by
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January 1990

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Foreword

These reports form the final output of the Phase I Coal Processing Development Consortium that jointly pursued the funding and development of advanced coal/oil agglomeration technologies from June 1987 to December 1989. For many of the participants, it was a challenging new experience and one that led to some important lessons and technical opportunities. Technology resulting from the treatment of tarry wastes is being seriously pursued as a commercial venture in Canada and the USA. The pyrite rejection technology is being scaled up in the USA, and the low rank coal/heavy oil technologies are being investigated in Canada. The financial and technical support of the consortium members in Phase I is acknowledged and recognized. Without their support, the program would not have been as successful as it was.

Phase II of the Coal Processing Development Program will be continued until December 1990 to provide more in-depth engineering data and scale up support for the key program elements. Results of Phase II and the overall conclusions will be reported

in 1991. The continuing support of the Phase II participants is also acknowledged, and they are thanked for their on-going interest and contributions.

The Alberta Research Council thanks the Electric Power Research Institute, their program staff and representatives on the management and technical committees, for their support and guidance in our mutual work. Our special thanks go to Howard Lebowitz, Linda Atherton, Conrad Kulik, Bill Rovesti, Bill Weber and Norm Stewart.

Finally, we wish to thank the technical and support teams of the Alberta Research Council who have made the projects as successful as they are and who have worked under ever increasing demanding scopes of work as the program developed and improved.

Dr. Arvid H. Hardin, Department Head
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Devon, Alberta
July 1990

Abstract

This report documents work completed by the Alberta Research Council for the ARC/EPRI Consortium, under the program entitled "Coal Processing Development Program: Development of Clean Coal and Clean Soil Technologies Using Advanced Agglomeration Techniques". The report is divided into three volumes:

Volume 1. Upgrading of Low Rank Coals: The Agflotherm Process

Test data, procedures, equipment, etc., are described for co-upgrading of subbituminous coals and heavy oil. The test results showed upgraded coals to have heating value above 11,000 BTU/lb, and syncrude (upgraded heavy oil) to have 21° API gravity or better. The coal products were acceptable for combustion and had handling properties superior to those of subbituminous coals. The syncrude was suitable for pipelining and refining. The techno-economic feasibility study of the Agflotherm process suggests that upgraded coal could be produced at \$Cdn 37.90 per metric tonne at 15 percent DCF ROR (fob plant, after tax).

Volume 2. Upgrading of Bituminous Coals: The Agfloat Process

Experimental procedures and data, bench and pilot scale equipments, etc., for beneficiating bituminous

coals are described. The test results showed the beneficiated coal to have heating value of 13,000 BTU/lb or better, ash content of less than 10 percent and pyritic sulphur removal of up to 90 percent. The completed techno-economic feasibility study of the Agfloat process suggests that beneficiated coal could be produced at \$U.S. 52.85 per short ton, at ROM coal prices of \$U.S. 27/t. For coal wash plant wastes, the product price would be \$U.S. 25.26 per ton.

Volume 3. Soil Clean-up and Hydrocarbon Waste Treatment Process

Batch and pilot plant tests are described for soil contaminated by tar refuse from manufactured gas plant sites. The test results show the treated soil to be suitable for disposal in landfills and the by-product agglomerates containing tar and hydrocarbon contaminants to be suitable for combustion in industrial boilers. The techno-economic feasibility study of a mobile plant for the Clean Soil process shows the cost of cleaning to range from \$Cdn 31.39 to \$Cdn 46.97 per tonne of oily wastes, and to be about \$Cdn 45 per tonne of tar refuse contaminated soil.

Acknowledgements

The multidisciplinary and diverse nature of the program undertaken required a pool of experts from several fields of coal development technologies. As a result, the achievements described in this report are, in part, also due to the contributions of several subcontracted companies and individuals. Specifically, we would like to acknowledge the contributions of Combustion Engineering Inc., Hartford, Connecticut,

for combustion and agglomerates handling tests; Sam Wong of ARC Technical and Economic Evaluation Group, for economic analysis and costing of the conceptual mobile plant; Lobbe Technologies Ltd., Regina, Saskatchewan, for the preparation of this Final Report; and the Alberta Research Council's Corporate Communications department for the production of this Final Report.

Executive Summary

I. Introduction

In 1987, the Alberta Research Council and EPRI formed a research consortium to develop new clean coal and clean soil technologies based on ARC's advanced spherical agglomeration research. The Consortium included 19 companies, government agencies and non-profit research organizations (see Appendix 1). The Consortium identified three technology development areas as being of immediate interest to its members:

1. Co-upgrading of low-rank coals and heavy oils.
2. Beneficiation of high sulphur bituminous coals.
3. Cleaning of tar refuse and hydrocarbon contaminated soils.

The Consortium authorized ARC to undertake research and development programs addressing technology needs in each of the identified areas. The programs were to include laboratory research and testing, fundamental engineering studies, development and operation of integrated agglomeration pilot plant facilities, and conceptual design and economic studies for the processes developed. The following is a summary of the projects undertaken and work completed to December 1989. It should be emphasized that since December, 1989, major progress has been made in further development of the three technologies and particularly in contaminated soil clean-up.

II. Co-Upgrading of Low Rank Coals and Heavy Oils

The objective of the project was to apply advanced agglomeration technology for co-upgrading of low-rank coals and heavy oil, namely:

- to increase the heating value of low-rank coals above 25.5 GJ/t (11,000 Btu/lb) by dewatering and deashing (upgrading) low-rank coals to capacity moisture and ash content of less than 10 percent each; and
- to upgrade heavy oil and/or bitumen used in agglomeration of coal to synthetic crude which would be suitable for pipelining and acceptable to refineries.

The outcome of the study was the development of the Agfiotherm process (agglomeration, floatation and thermal treatment) which upgrades low-rank coal to high heating value solid fuel and heavy oil or

bitumen to synthetic crude oil. Specific process research achievements related to the Agfiotherm process are summarized below.

Batch Test Studies

Batch test procedures were developed for characterization of various coal and oil feedstock combinations for treatment in the Agfiotherm process. The tests defined agglomeration and de-oiling parameters important in process optimization and operation. Selected results and products obtained in the process are shown in Table I. The product agglomerates from subbituminous coals had properties similar to those of western Canadian bituminous coals, and product upgraded oils were similar to synthetic crudes produced from bitumen in other heavy oil thermal upgrading processes.

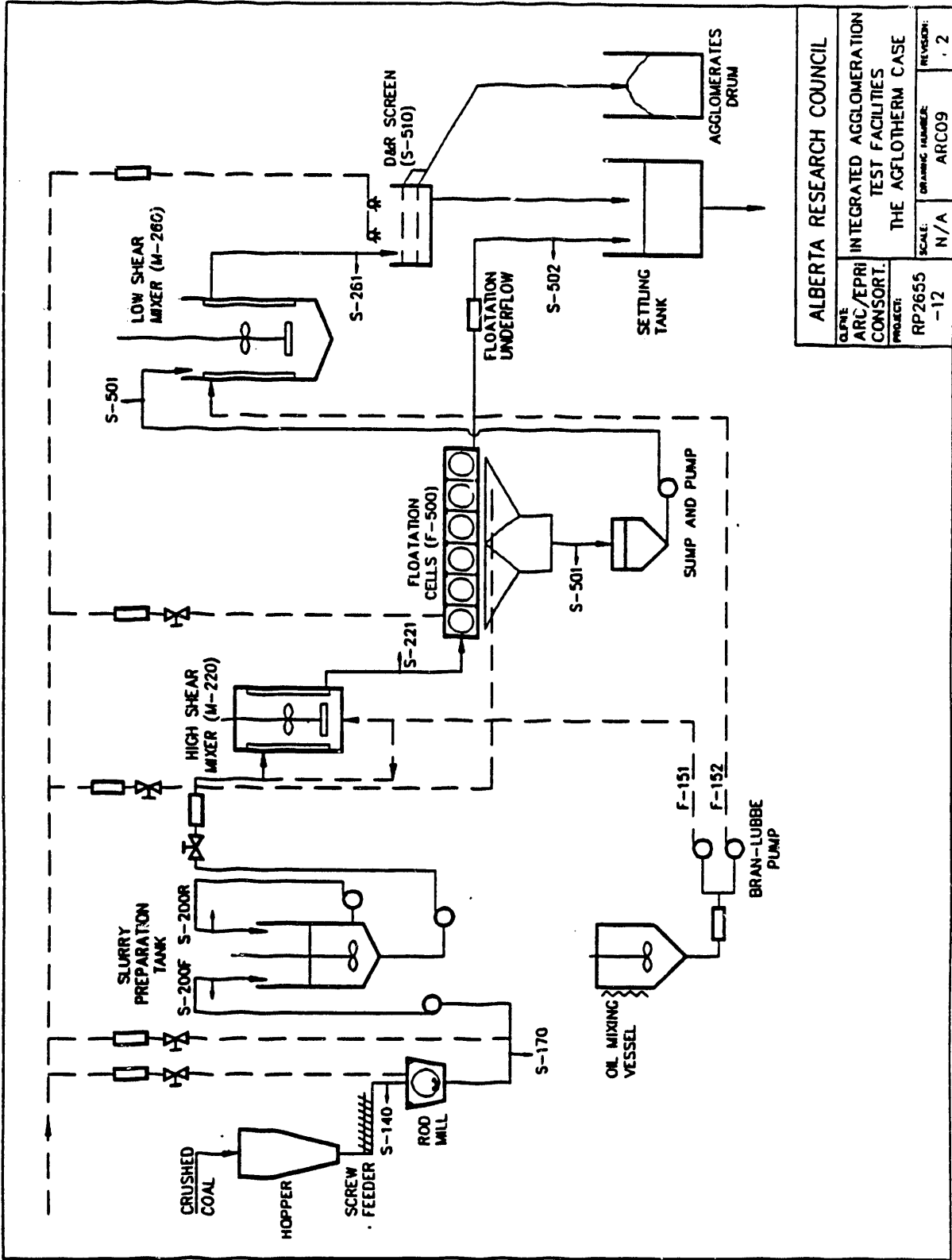
Table I. Upgrading of Low Rank Coal in Agfiotherm Process: Heatburg Coal, Batch Tests.

	Moisture, %	Ash, %	Volatile matter, %	Fixed carbon, %	Heating value Btu/lb
Feed Coal Green	16.3	24.6	30.3	28.8	7,840
Agglomerates	8.2	9.7	46.0	36.1	11,370
De-oiled Agglomerates (de-oiling temperature)					
350°C	4.1	10.8	35.8	49.3	11,580
390°C	3.5	12.5	31.5	52.5	11,530
420°C	3.5	13.9	24.6	58.0	11,320

Oil Recovery Studies

The bench scale tests showed that it is technically feasible to recover upgraded oil from coal agglomerates, and that oil yield and properties are attractive enough to consider potential commercialization of the process. The upgraded oil had API gravity above 21, and significantly reduced sulphur and nitrogen content. Different cuts from the upgraded oil blends were found to have acceptable properties for refining, namely:

- Naphtha cut (c₅/175°C) was rich in aromatics and had a higher octane number than petroleum-derived naphtha cuts. It would have to be hydrotreated before reforming (like all naphtha cuts from heavy oil syncrudes) and after treatment the blend could be used as a reformer feedstock for gasoline production.



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CLIENT:	ARC/EPRI	SCALE:	N/A
CONSORTIUM:	INTEGRATED AGGLOMERATION TEST FACILITIES	DRAWING NUMBER:	ARC09
PROJECT:	THE AGFLOTHERM CASE	REVISION:	. 2
	RP2655		
	-12		

- Jet fuel cut (145/260°C) had a low smoking point and would also require some hydrotreatment.
- Diesel fuel blend (177/343°C) would require hydrotreatment to reduce the contents of aromatics, sulphur and nitrogen.
- Vacuum gas oil cut (+343°C) had low content of Ni and V, but high sulphur and nitrogen.

Integrated Agglomeration Test Facility (IATF)

Engineering and operational data were obtained for the Agflotherm process using the IATF specially reconfigured for the process (flowsheet #ARC09).

The plant produced well-formed, large agglomerates; however, in processing some feeds, difficulties were experienced with obtaining satisfactory performance of the pilot plant. These difficulties were primarily due to insufficient experience and process information in areas such as:

- kinetics of agglomeration for coals characterized by long inversion times,
- optimization of the floatation conditions for microagglomerates obtained from low-rank coals which contained a high clay concentration,
- measurement of slurry flow, slurry solid concentration, density, etc., and
- process control system.

The pilot plant products were de-oiled and sent for laboratory combustion tests. The products were found to have good handling, storage and combustion characteristics including less susceptibility to spontaneous combustion than the "parent" coals.

Feasibility of the Agflotherm Process

A conceptual design for the Agflotherm process was developed to upgrade 2.8 million metric tonnes of ROM subbituminous coal and 448 thousand tonnes of heavy oil in Alberta, Canada. The plant production was estimated at 2.06 million tonnes per year of de-oiled agglomerates with a heating value of 26.7 GJ/t (11,300 Btu/lb) and 370 thousand tonnes of syncrude with API gravity of 21. The plant cost was estimated at \$Cdn 113.6 million, operating cost at \$Cdn 91 million per year and working capital at \$Cdn 17.2 million. Based on 1989 feedstock and product prices (\$Cdn 12/t of coal, \$Cdn 93.4/t bitumen, \$Cdn 33/t product agglomerates and \$Cdn 124.2/t syncrude), the economic analyses of the plant showed DCF ROR after tax equal to 11.1 per cent.

The key cost factors in the Agflotherm were the cost of heavy oil and coal, the yield and price of co-product oil, and the price of de-oiled agglomerates. The projected DCF ROR of 11.1 percent was contingent on the technical feasibility of a novel de-oiling process based on high temperature extrusion of coal-oil paste (described in Volume 1).

III. Beneficiation of High Sulphur Bituminous Coals

The objective of the bituminous coal project was the development of technology which would:

- remove pyritic sulphur from high sulphur bituminous coals,
- Reduce mineral matter content in bituminous coals to 10 percent or less, and
- increase heating value of the processed coals above 13,000 Btu/lb.

The result of the process research was the development of a process, the Agfloat process (agglomeration and floatation), capable of meeting all of the above listed project objectives. Specific process issues related to the performance and economics of the Agfloat are discussed below.

Batch Test Studies

Laboratory tests were carried out to evaluate key factors influencing the Agfloat process performance. Nine coals (ROM and coal preparation by-products) from the eastern U.S. and western Canada were studied. Typical results obtained with U.S. coals are shown in Figure 1. The results were obtained using single-stage and two-stage (with regrinding and reprocessing of the product) batch processing. The tests show good combustibles recovery and pyrite rejection at bridging oil concentrations as low as 0.5 percent. For western Canadian coals, however, the Agfloat procedures had to be modified and the amount of bridging oil had to be increased to 3 percent or more.

Continuous Pyrite Removal Unit (CPRU)

The feasibility of using hydraulic and flume separators for removal of pyrite from bituminous coals agglomerates was evaluated using a 5 kg/h bench scale CPRU. Both units showed reduction of pyrite in coal product; however, because of better

operability, a system with a hydraulic separator only was chosen for the Agfloat process. Tests using the CPRU with U.S. coals indicated that at the best conditions tested, the total sulphur reduction ranged from 50 to 55 percent, and the pyritic sulphur reduc-

tion ranged from 72 to 77 percent, while the ash reduction ranged from 82 to 86 percent. The information obtained in the CPRU was successfully used in the scale-up of the hydraulic pyrite separator in the pilot plant (IATF).

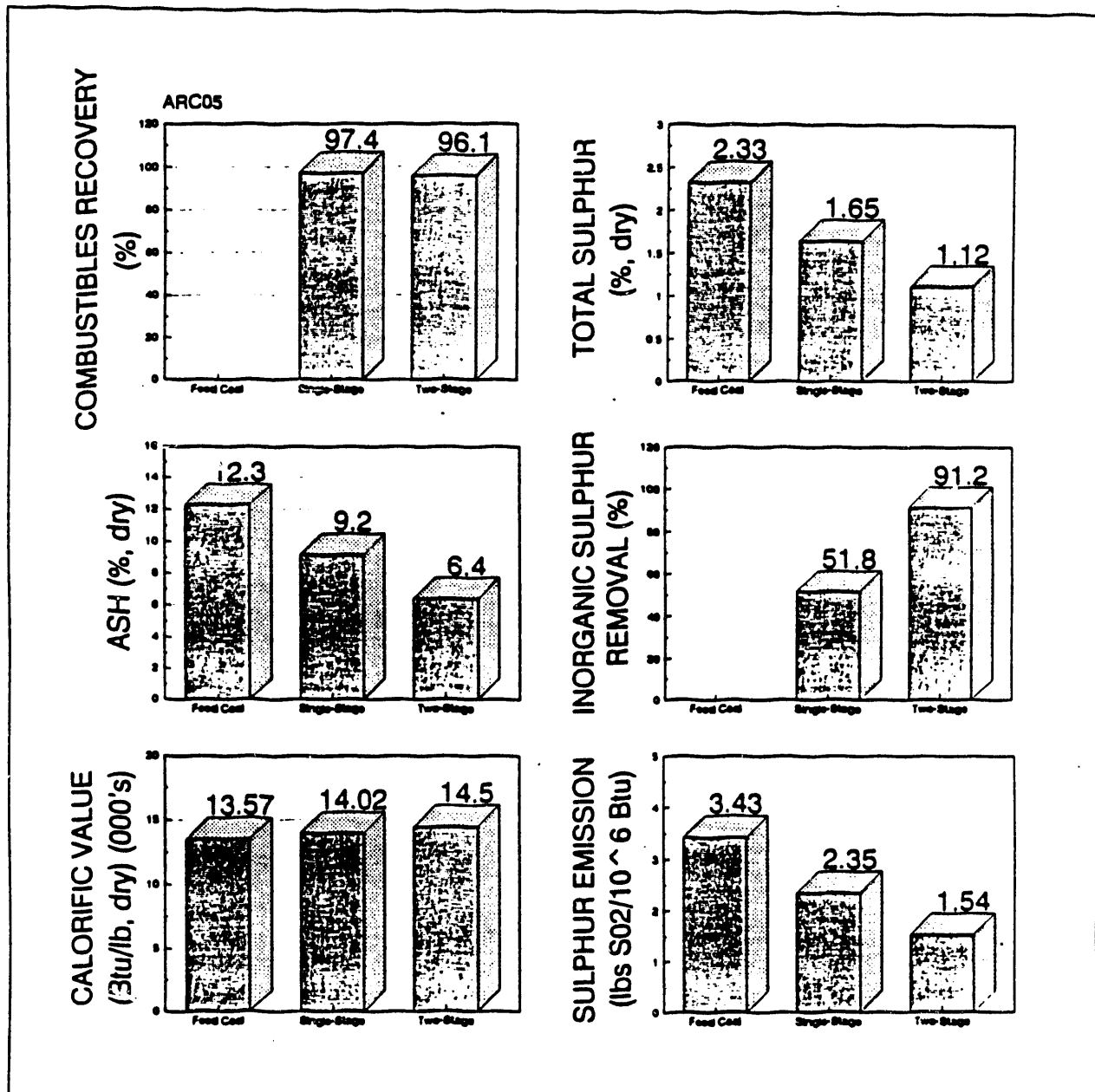
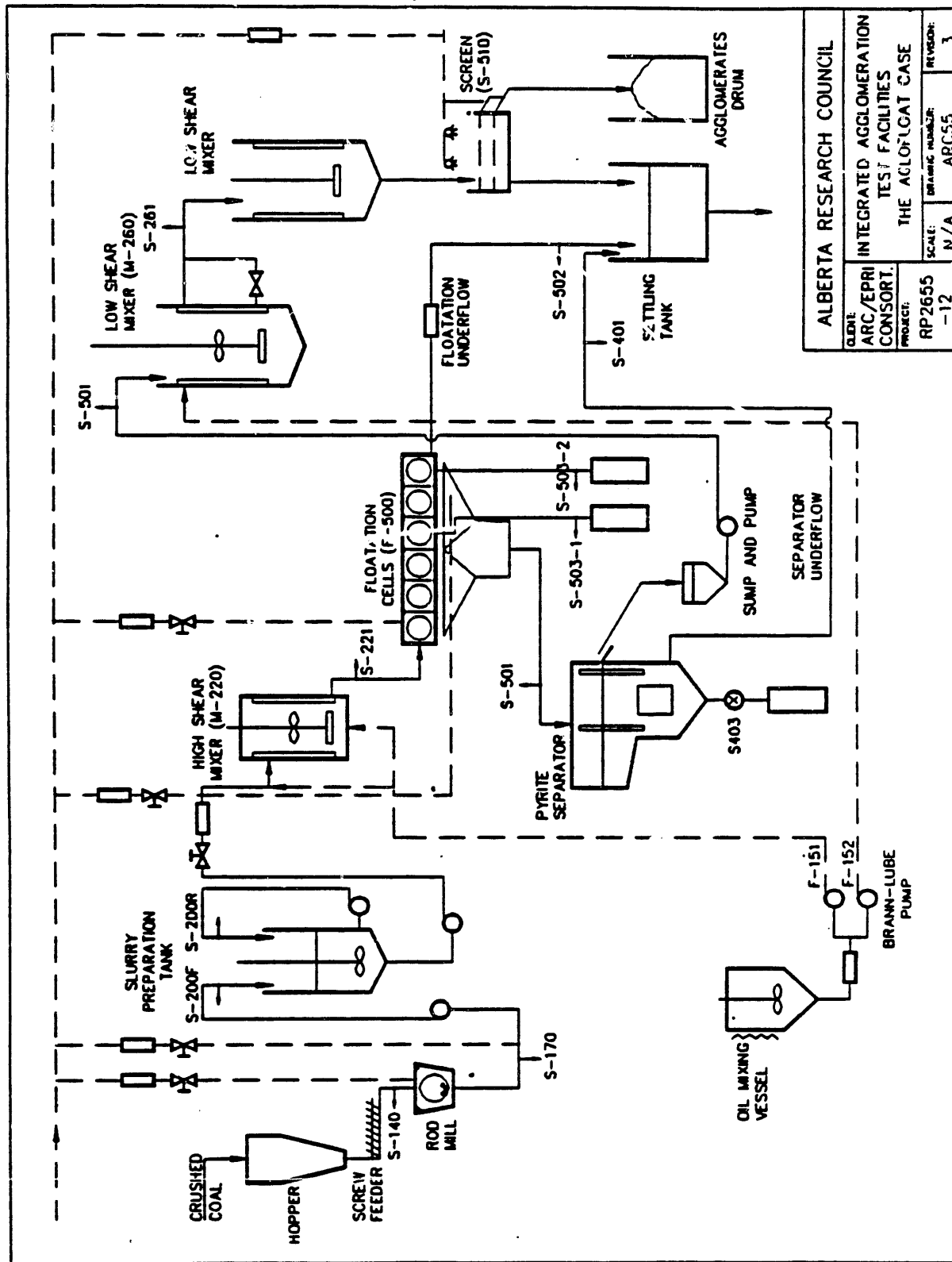


Figure 1. Cleaning of Upper Freeport Coal by Agfloat Process.



ALBERTA RESEARCH COUNCIL			
CLIENT:	ARC/EPRI INTEGRATED AGGLOMERATION TEST FACILITIES		
PROJECT:	THE AGGLOMGAT CASE		
SCALE:	N/A	ARC55	REVISION: 3
PROJECT NUMBER:	RP2655		
	-12		

Large Pilot Plant Tests (IATF)

The hydraulic pyrite separator developed was incorporated into the Integrated Agglomeration Test Facility pilot plant in the Aglofloat process configuration (flowsheet #ARC55). The IATF results confirmed that well-formed agglomerates reduced in sulphur and mineral matter can be obtained in the process. Large, 0.8 to 3.0 mm agglomerates could be obtained at bridging oil concentrations as low as 12 percent (d.a.f. feed coal). The agglomerates had a heating value above 13,000 Btu/lb, ash content of less than 10 percent and pyritic sulphur content reduced up to 90 percent.

Considerable effort went into improving the process. Equipment efficiency and performance was evaluated for unit operations such as floatation of coal flocs, hydraulic pyrite separation and size enlargement of microagglomerates. One of the key issues studied was the effect of process operating parameters on process stability and performance. The key Aglofloat process parameters were identified; however, more experimentation will be necessary to understand the process and to further reduce the amount of bridging oil required.

Feasibility of the Aglofloat Process

The pilot plant work for the Aglofloat process was used as a basis for the conceptual design of an aglofloat plant capable of processing 2.8 million short tons of eastern U.S. bituminous coal to 2.74 million tons of agglomerates. The economic analysis of the process estimated the plant capital cost at \$U.S. 61.5 million, operating cost at \$U.S. 121.0 million per year and working capital at \$U.S. 22.2 million. The product cost was calculated at \$U.S. 52.85 per ton at DCF ROR equal 15 percent (after tax) and coal price of \$U.S. 27/t FOB plant. The key cost elements determining the product price were the coal price, the amount and price of oil, and to a lesser extent the operating and capital costs.

Variance analysis performed on the coal price suggested that using the Aglofloat to process coal refuse (coal price = \$U.S. 0/t) could produce a clean coal product at \$U.S. 25.26 per ton. Such a product would be competitive with the current cost of low- and medium-sulphur coals in the eastern U.S. (priced at \$U.S. 20 to \$U.S. 28 per ton).

IV. Cleaning of Tar Refuse and Hydrocarbon Contaminated Soils

The objective of the Clean Soil study was to develop technology which would:

- clean up contaminated soils at manufactured gas plant sites, coking plant sites, benzol plant sites, etc.;
- clean up contaminated soils from oil spills, oily wastes pits, and other hydrocarbon contaminated spills; and
- treat heavy oil wastes and emulsions from heavy oil recovery and upgrading plants.

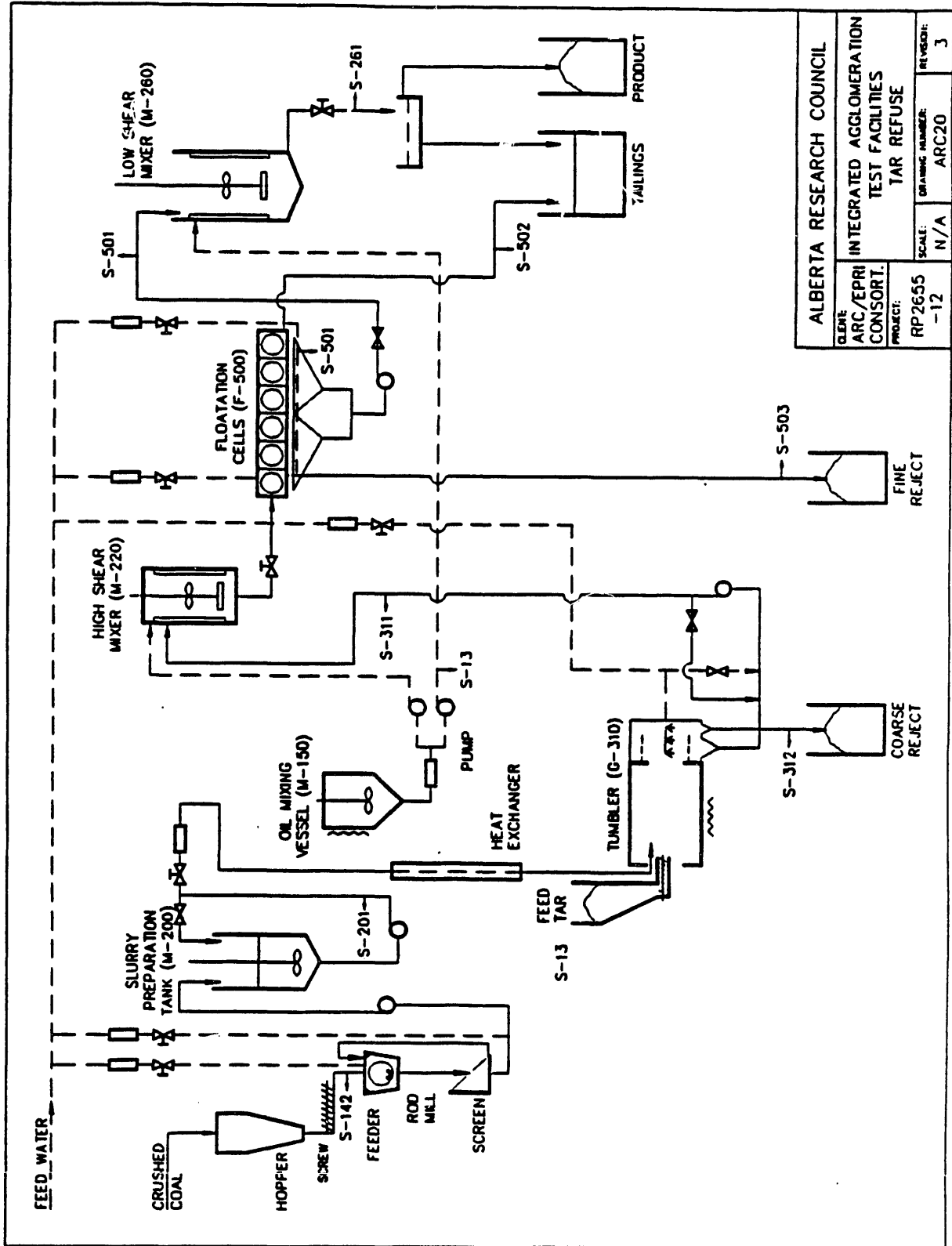
The project led to the development of the Clean Soil process which cleans contaminated soils to a level of 1000 ppm (0.1 percent) or less, and yields by-product agglomerates which contain the hydrocarbon contaminants and are suitable for combustion in industrial boilers. Specific achievements related to the process performance and evaluation are summarized below.

Batch Test Studies

A rapid laboratory screening method for characterization of various contaminated soils for treatment in the Clean Soil process was developed. The tests performed suggest that a wide range of contaminant types and concentrations in the soil can be treated by the process. The concentration of residual contaminants in the clean soil was reduced to 0.1 percent or less (Table II). The key properties of the contaminated soil influencing the process performance were identified as:

- the amount and composition of the hydrocarbon contaminants;
- the particle size and particle size distribution of the soil; and
- the presence of other materials in the contaminated soil capable of adsorbing the contaminants, e.g., coke.

Depending on the soil characteristics, the Clean Soil process procedures can be readily modified to achieve the best process performance.



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CLIENT:	INTEGRATED AGGLOMERATION TEST FACILITIES		
PROJECT:	TAR REFUSE		
SCALE:	N/A	DRAWING NUMBER:	ARC20
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Table II. Clean-up of Contaminated Soil Using Clean Soil Process: Overall Material Balance for Site #1-1, Batch Tests.

Type of contaminant	Contaminant concentration, %	
	Feed	Processed soil*
Tar #1(1)	8.6	0.07
Tar #1(2)	1.2	0.00
Tar #2(2)	66.9	0.10
Tar #2(8)	0.7	0.10
Tar #1-1	5.6	0.07
Tar #2-2	10.8	0.17
Heavy Oil	8.7	0.04
Oil (light) Spills	2.0	0.17
Gasoline Spills	3.1	0.06
Oil (heavy) Spills I	43.0	0.06
Oil (heavy) Spills II	0.2	0.00

* Guidelines accepted by many regulatory agencies in North America require that the residual concentration of petroleum derived contaminants does not exceed 1000 ppm (0.1 percent) in the clean soil.

Large Pilot Plant (IATF)

The Integrated Agglomeration Test Facility was reconfigured for the Clean Soil process operation (flowsheet #ARC20). The pilot plant runs provided information on process control and performance of each unit operation at steady-state conditions. For some feeds, however, less than desired performance of the IATF was obtained because limited process information was available on the kinetics of agglomeration and the separation of contaminants from the cleaned soil.

A large sample of product agglomerates produced in the IATF was tested for its handling, grinding and combustion characteristics by Combustion Engineering, Inc. The tests showed that combustion performance and emissions for agglomerates from tar refuse were similar to combustion performance of HVB coal used in agglomeration.

Feasibility of the Clean Soil Process

The commercial feasibility of using the Clean Soil process for treatment of oily waste from bitumen upgrading was evaluated. The plant was assumed to be a mobile plant located in Alberta and capable of processing 30,500 tonnes of oily wastes per year. Depending on whether pulverized coal was purchased or prepared at the plant site, the plant capital cost was estimated at \$Cdn 1.8 and \$Cdn 2.06 millions, respectively. The cost of cleaning oily waste was estimated at \$Cdn 31.39/t, for a case where the

market for agglomerates exists near the plant, and at \$Cdn 46.97/t for a case where agglomerates would have to be shipped for sale. The costs indicated are within the range of current costs of waste disposal (\$Cdn 25 to \$Cdn 35 per ton, for hauling and spreading). However, since spreading is likely to be unacceptable as a disposal method in the future, the Clean Soil process offers a viable new technology for oily waste disposal.

V. Conclusions

Three new processes were developed in the first two and one-half years of the Consortium Coal Processing Development Program based on ARC's advanced spherical agglomeration research. The preliminary evaluation of these processes suggest that the most promising opportunities and their constraints for further development, are:

- co-upgrading of low rank coal and heavy oil,
- beneficiation of bituminous coal refuse and ROM coal, and
- clean-up of contaminated soil.

The major technical and economic constraints which must be solved for each process are:

- the technical feasibility of coal-oil paste extrusion in co-upgrading of coal and heavy oil,
- the reduction of the amount of bridging oil required for production of large size agglomerates in beneficiation of bituminous coals, and
- the optimization of the reprocessing steps in cleaning of some contaminated soils.

The results described in Volumes I, II, and III of this Report summarized achievements completed prior to December 31, 1989. Since that time, however, further improvements in process performance were attained and presented to the Consortium members. These results will be described in detail in the updated reports for each process.

Each of these technology opportunities has advanced in this year (1990) of the consortium program, to a stage where it can be pursued by smaller R&D consortia comprised of members with an interest in one or two of the technology areas only. It is recommended that future work in this program be pursued by such consortia addressing each technology area separately.

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1. Introduction

1.1. The Clean Soil Process

The Clean Soil process is a spherical agglomeration process capable of cleaning contaminated soils from manufactured gas plant sites, oil spills and oily waste pits. In the process coal is agglomerated using the oily/tarry waste in the sand refuse as bridging oil. The products of the process treatment are clean soil suitable for disposal in land fills and solids fuel made up of the coal and hydrocarbon contaminants, suitable for combustion in industrial boilers.

A simplified schematic of the process and product streams is given in Figure 1.1. The depicted process configuration is for a mobile soil treatment plant capable of processing oily and tarry wastes.

The clean-up process starts with contaminated soil being fed into the tumbler where it is mixed with hot coal/water slurry. The contents are then screened and the -1 mm material is transferred to a high shear mixer where soil cleaning and the growth of fuel particles continues. The microagglomerates (fuel products) are separated from the clean soil in a flotation cell. Larger agglomerates (>1.0 mm) can be produced by further processing of the microagglomerates in a low shear mixer. The green agglomerates are screened and air dried in stockpiles. The fine and coarse clean soil are combined, filtered and disposed of.

The Clean Soil process offers several advantages for treatment of contaminated soils and oily wastes, namely:

- It is applicable to a wide range of contaminants and contaminants' sites.
- It meets stringent product specifications for cleaned soils.
- It has a lower cost and better energy efficiency relative to incineration based processes.
- It is simpler than the extraction processes and does not require expensive solvents.
- It promises to be the most cost effective process overall for the above reasons.

The process is effective for clean-up of soils having tar and/or oil concentrations from as little as 1 percent to over 50 percent. It can be operated at contaminant/coal ratios from a few percent to 40 percent, and contaminated soils which contain coarse and porous materials can be cleaned with minor process modifications.

1.2. Objectives and Scope of the Program

The overall objective of the Coal Development Program has been described in detail in Section 1.2, Volume 1 of this report. The general objectives with respect to contaminated soil clean-up have been the development of technologies which would:

- clean up contaminated soils at manufactured gas plant sites, coking plant sites, and benzol plant sites;
- clean up contaminated soils from oil spills, oily wastes pits, and other hydrocarbon spills; and
- treat heavy oil and emulsion spills from heavy oil recovery and upgrading plants.

The specific objectives of the Clean Soil program were to explore and evaluate the application of the agglomeration technology for:

- clean-up of contaminated soil from manufactured gas plant sites by removing the soluble tar;
- recovery of combustible material from tar refuse; and
- testing the handling and combustion properties of the recovered coal/tar agglomerates.

The project addressed a number of research issues related to establishment of the technical and commercial viability of the Clean Soil process, e.g., solution of engineering problems, and improvement of process efficiency and economics. In addition, the cleaned soil had to be shown to be acceptable for disposal and the coal/tar oil agglomerates had to be shown acceptable for combustion in industrial boilers.

The key technical and process economic areas addressed in the project were:

- **Process Research Studies:** to determine the expected process performance using batch characterization methods for selected feeds, and to obtain process information useful to the design and operation of the Integrated Agglomeration Test Facility (IATF).
- **Engineering Studies:** to determine the key design concerns for the IATF and commercial plants, to develop process control strategy, to obtain engineering and operating data required for process evaluation, and to develop design criteria for conceptual design and cost analysis of the Clean Soil process.
- **Product Characterization:** to assess the combustion behavior of the product agglomerates and to determine their handling and storage properties.

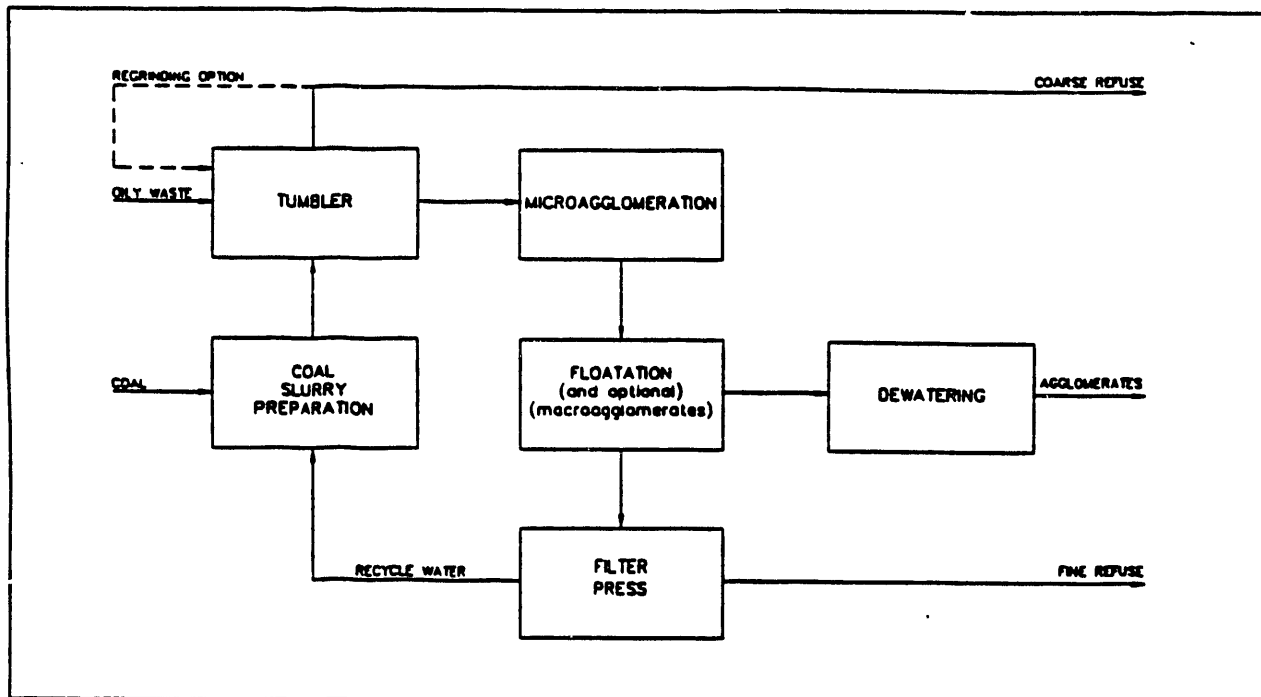


Figure 1.1. Schematic representation of the Clean Soil process.

- **Economic Studies:** to determine the economic feasibility of the process including variance analysis with respect to major economic and process parameters.

The scope of the project covered a number of diverse activities including batch and continuous scale agglomeration and separation tests, construction and operation of a 6 tonne per day pilot plant, and characterization of product agglomerates in bench and pilot scale combustion rigs.

1.3. Project Organization

The project organization chart is presented in Figure 1.2.

The project was overseen by two committees: 1) a Management Committee with responsibilities for program planning and funding, and 2) a Technical Committee with responsibility for program operation and conduct. The details of the specific responsibilities of both committees are provided in Section 1.3, Volume 1 of this report.

1.4. Report Organization

The results of the ARC/EPRI Coal Processing Development Program are presented in three volumes, each addressing specific applications of advanced agglomeration technology:

Volume 1 – Upgrading of Low Rank Coals: The Agglomer Process

Volume 2 – Removal of Ash and Pyritic Sulphur from Bituminous Coals: The Agfloat Process

Volume 3 – Soil Clean-up and Hydrocarbon Waste Treatment Process: The Clean Soil Process

Each volume of the report is supplemented by an Appendix containing relevant background data and additional information and descriptions of process designs.

This volume presents the results of ARC's laboratory and pilot plant work on the development of improved technology for soil clean-up and hydrocarbon waste treatment. Chapter 2 begins with a description of contaminated soils selected for testing with the Clean Soil process. The chapter includes descriptions of the bench experimental procedures used and the results of process research and development work.

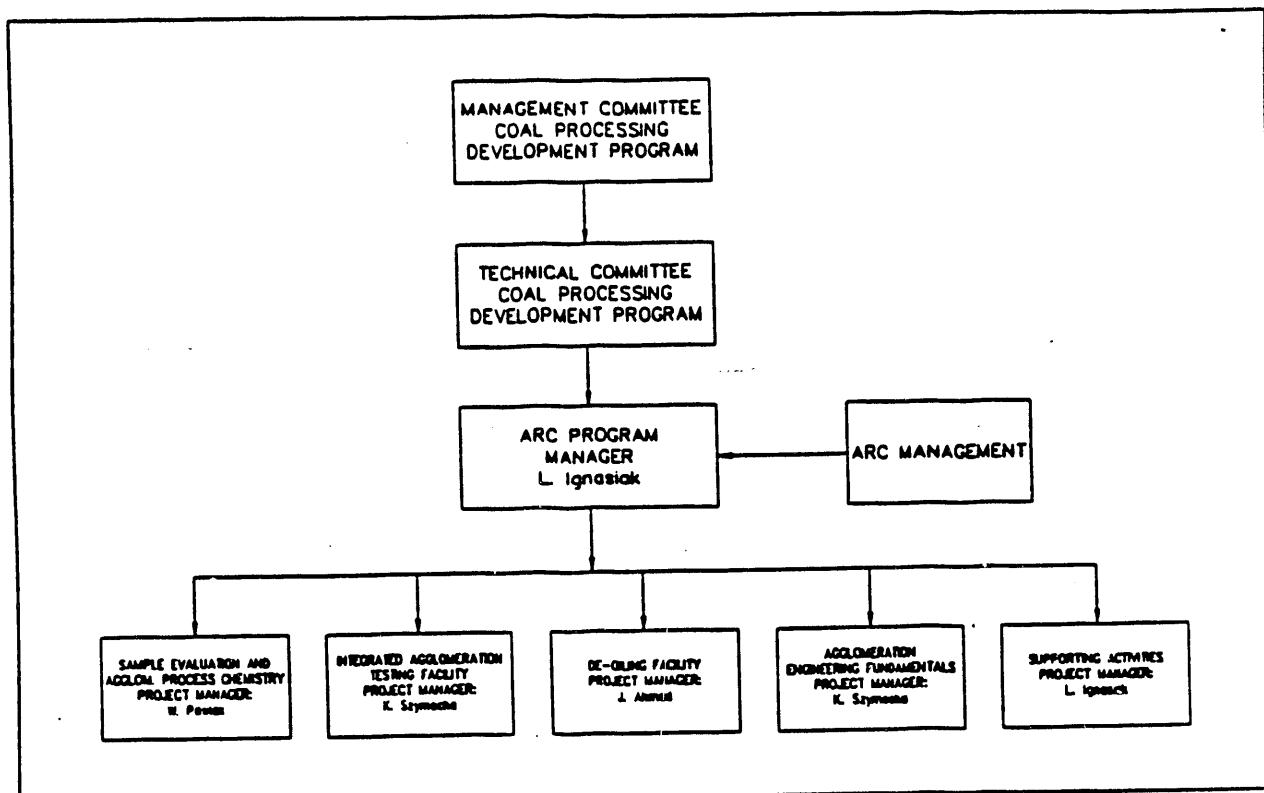


Figure 1.2. Project organization chart.

In Chapter 3, Volume 3, design studies of the agglomeration mechanisms are discussed. These include evaluation of critical design issues in the process.

Chapter 4 describes the experimental results from the Integrated Agglomeration Test Facility (PDU). The chapter starts with a review of major equipment and instrumentation. This is followed by a description of the plant commissioning and details of the operating experience.

Chapter 5 includes a description of the pilot-plant combustion tests, and handling and storage tests conducted on agglomerates produced from tar refuse. The description includes discussion of general grinding characteristics, fireside performance, etc., of the agglomerates.

Chapter 6 reviews environmental regulations which are likely to apply to the operation of a mobile Clean Soil plant for cleaning oily wastes from a bitumen extraction plant in Alberta. It lists environ-

mental regulations and compliance required depending on the application of the process.

The conceptual design and cost analysis for a mobile Clean Soil plant are presented in Chapters 7 and 8. The information presented includes process and economic data and financial analysis of the plant operated to clean oily wastes from the bitumen recovery plant in Chapter 7 and process and economic data for the plant operated to clean tar refuse from manufactured gas plant sites in Chapter 8.

Chapter 9 presents the major conclusions and recommendations relating to the Clean Soil process. The key issues addressed are further improvements of the process efficiency, and development of a broader data base for cleaning different soils. The references quoted in Volume 1 of the report are presented in Chapter 10.

2. Bench Scale Characterization of Contaminated Soils

2.1. Experimental Procedures

2.1.1. Approach

The batch tests leading to development of the Clean Soil process had the following objectives:

1. To determine the composition of tar refuse samples submitted in the program.
2. To assess the suitability of tars and other hydrocarbons in the tar refuse samples for use as a bridging oil in agglomeration.
3. To obtain process research information useful for the design and development of the Aglofloat process for clean-up of tar refuse.
4. To develop data and information required in order to use the Integrated Agglomeration Test Facility for cleaning tar refuse samples.

Each test used up to 550 grams of contaminated tar refuse, 250 grams of coal and an appropriate amount of water. The size of a total charge used in the batch experiments was determined by the capacity of the equipment.

Figure 2.1 provides a schematic representation of the experimental procedures used for batch evaluation of tar refuse samples. The experiments were performed in five steps. First, tar refuse was mixed with a slurry of coal and water in a 2 liter tumbler rotating at 60 rpm, for 5 to 15 minutes, at selected

temperatures in the 70° to 80°C range. The bituminous coal used was pulverized to a nominal top size of 600 Mm (-0.6mm). Next, all coarse material with a particle size larger than 3.3 or 1.0 mm was removed by screening. In the third step, microagglomerates were formed by high shear mixing and then separated from the slurry by froth floatation in a standard DENVER floatation machine. In the fourth step, the froth and tailings were dried (filtered and washed first for tailings) at 60°C for 4 hours and weighed. Finally, the products were extracted with dichloromethane to determine their tar content.

2.1.2. Equipment

The batch test equipment included a tumbler, a laboratory floatation machine and auxiliary equipment and instrumentation used for sample analyses, product analyses, handling, etc.

The tumbler was a 2 liter, double wall stainless steel vessel with four baffles. The space between the walls was filled with sand to maintain the contents' temperature.

The laboratory floatation machine was a model D-12 DENVER floatation cell with interchangeable impeller and diffuser parts. The machine produces its own air but an external, pressurized floatation air

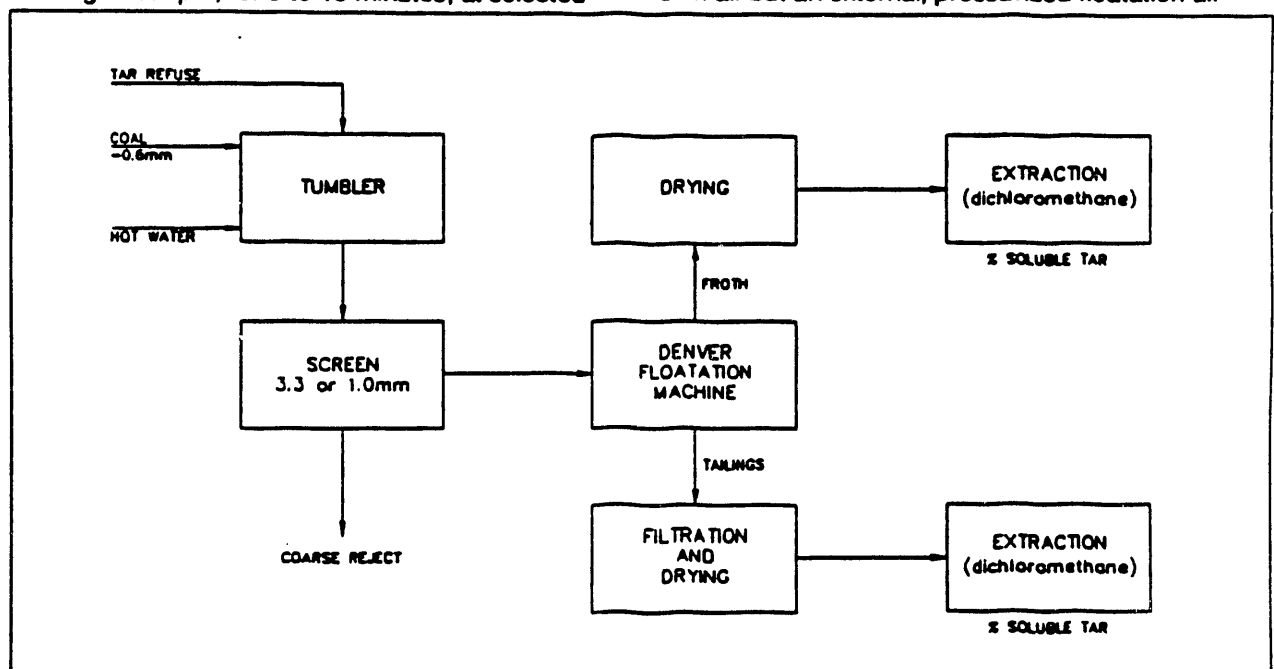


Figure 2.1. Schematic representation of batch testing procedures.

source could be used. The machine could be used as a rough separation cell or as a laboratory floatation apparatus for studying the floatation characteristics of various materials. The machine could also be used for studying the effects of various flocculents and additives on the recovery of hydrocarbons from the tar refuse samples.

2.1.3. Sample Preparation

Several small grab samples and two large bulk samples of contaminated soil were submitted for testing in the Aglofloat process. All samples were prepared for the batch tests as follows:

- Small grab samples were visually assessed for homogeneity and water content and, where required, samples were mixed by rocking, tumbling or stirring.
- Representative subsamples were taken from the homogenized tar refuse to determine the tar and water content of each grab sample submitted.
- Large rocks were removed.
- Samples submitted in drums were blended in a cement truck and redistributed to drums again after removal of large rocks and other attrital

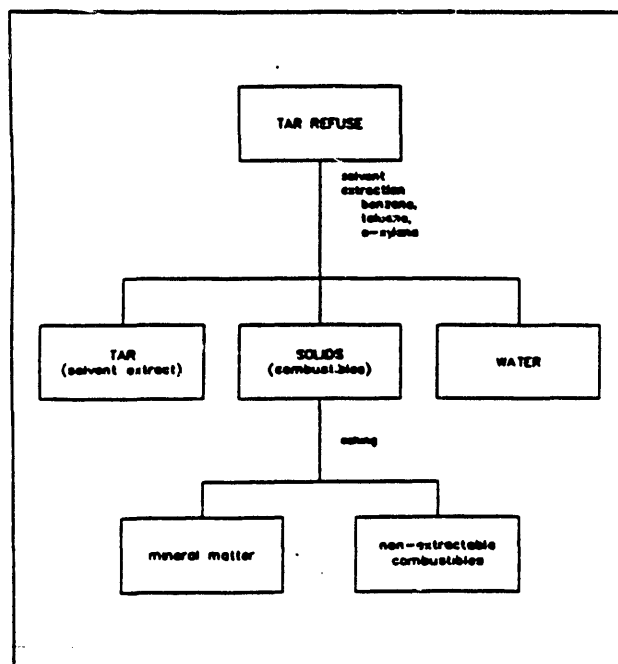


Figure 2.2. Procedures for determination of composition of tar refuse samples.

materials. Small subsamples were taken from drums for analyses.

2.1.4. Test Methods and Procedures

Table 2.1 and Figure 2.2 depict the test methods and separation procedures used to characterize the tar refuse, coal and process products. Where required, other specialized tests were also employed to determine the structural characteristics of tars.

In general, evaluation of tar refuse samples involved a cleaning step where tar refuse was vigorously mixed with coal slurry and a floatation step where the combustible product in the form of microagglomerates was separated from the clean soil (tailings). The products of aglofloatation were tested for:

- ash and tar in clean soil (tailings)
- ash and tar content in combustible product.

Table 2.1. Test methods and procedures.

Sample	Test method	Procedure
Tar Refuse	Composition % water % tar % solids	Dean Stark Extraction with toluene
	Non-extractable combustibles, %	Proximate (Fisher Coal Analyzer; ASTM draft)
	Particle size	Screen analysis
Tar	Elemental comp. Volatility	Ultimate GC simulated distillation TGA
	Molecular weight distribution	Gel permeation chromatography (gpc)
	Hydrocarbon class analysis	Column chromato- graphy, infrared spectroscopy
	Component identification	Gas chromatography/ mass spectrometry (gc - ms) (EPA method SW 8240 and 8270)
Products: Combustibles (microagglom)	Composition % Tar % Combustibles % Ash	Solvent extraction (CH ₂ Cl ₂) Proximate Proximate
Clean Soil	Composition (see above) Particle Size	See above Screen analysis

2.2. Product Research and Optimization

2.2.1. Tar Refuse Samples Data

Sample Analyses

The objective of the analyses was to evaluate the properties of the tar refuse samples which were to be processed in bench-scale units. In total, twelve small grab samples and five bulk samples from two contaminated sites were submitted for testing.

The average composition of the grab samples is given in Table 2.2.

Composition histograms for the grab samples are given in Appendix 2, Figures A2.1 and A2.2.

For bulk samples, a list of the number of drums received from each site is presented in Table 2.3. The composition of the blended tar refuse samples for selected drums is presented in Appendix 2. An average composition for each site is presented in Table 2.4.

The range of composition for different tar refuse components was:

Table 2.2. Composition of tar refuse grab samples.

Source/ sample	Tar	Tar refuse, wt %		Ash in solids
		Solids	Water	
Site #1(1)	6.9	73.6	19.5	69.8
(2)	1.1	94.1	4.8	98.0
(3)	5.2	90.3	4.5	92.3
(4)	1.2	44.8	54.0	85.5
Site #2(1)	2.0	87.0	11.0	68.0
(2)	38.3	18.9	42.8	25.4
(3)	59.6	28.6	11.8	10.1
(4)	41.7	43.9	14.4	29.6
(5)	0.5	79.0	20.5	64.8
(6)	0.6	92.3	7.1	94.7
(7)	5.2	83.3	11.5	72.6
(8)	0.6	87.4	12.0	81.6

Table 2.3. Blending and screening of bulk tar refuse samples.

Source	Number of barrels	
	as received	blended*
Site #1-1	40	40
Site #1-2	30	26
Site #1-3	9	5
Site #2-2	125	81
Site #2-A	36	31

* After mixing and screening to remove large rocks.

Tar, % w/w	0.2 to 59.6
Non-extractable	1.9 to 30.9
Mineral matter, % w/w	2.9 to 92.2
Water content, % w/w	4.5 to 54.0

The key features depicted are:

- Samples #1(1), #1(4), #2(1), #2(5), #2(7), #2(8), and #2-2 have low and medium tar content yet very high combustibles.
- Samples #2(5), #2(6), #2(8), and #2-A all have very small amounts of soluble tar.
- Samples #2(2), #2(3), and #2(4) have very low mineral matter content.

Soluble Tar Analyses

A series of detailed analyses of soluble tar were conducted in support of refuse characterization. The analyses performed are listed in Table 2.1.

In this work, the term "tar" was defined as any organic material extractable with toluene and/or dichloromethane. In two instances, after removal of the tar with toluene, the solids were extracted with tetrahydrofuran (THF). The diagram of procedures is shown in Figure 2.3. The purpose of this test was to estimate the amount of high polar heteroaromatic compounds that might adversely affect the leachability tests. Such tests could eventually be required to comply with the Clean Air Act regulations.

The elemental composition, molecular weight distribution, simulated distillation, hydrocarbon type analysis, thermogravimetric analysis and infrared spectra are presented in Tables 2.5 to 2.10 and Figures 2.4 to 2.9.

In general, tar in contaminated samples was very aromatic. The nitrogen content was relatively low. Tar consisted of components characterized by a broad molecular weight distribution. Depending on the sample, the portion of tar with a molecular

Table 2.4. Average composition of blended bulk tar refuse samples.

Source	Tar	Tar refuse, wt %		Ash in solids
		Solids	Water	
Site #1-1	4.6	79.6	15.8	84.2
Site #1-2	1.0	83.3	15.7	-
Site #1-3	2.3	77.1	20.6	-
Site #2-2	8.9	75.3	15.8	66.2
Site #2-A	0.2	81.8	18.0	-

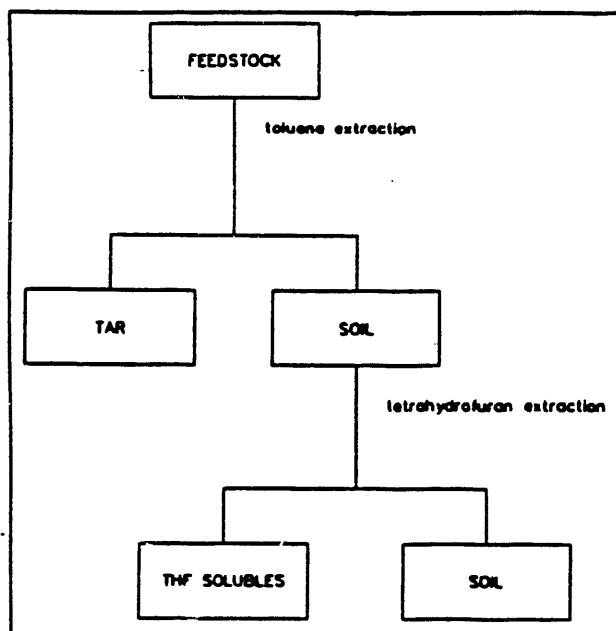


Figure 2.3. Diagram for extraction of soluble tar (tar and THF solubles).

Table 2.5. Elemental composition of tar.

Wt %	Grab sample #		Bulk sample # ^a	
	Site #1(1)	Site #1(3)	Site #1-1	Site #2-2
Carbon	91.1	90.8	83.8	84.3
Hydrogen	6.6	6.4	6.4	6.2
Nitrogen	<0.1	0.2	0.2	0.3
Sulphur	1.3	1.2	5.2	6.9
Oxygen	0.9	1.6	4.4	2.3
Atomic H/C	0.87	0.85	0.93	0.88

^a after bulk blending.

Table 2.6. Elemental composition of THF solubles.

wt, %	Bulk sample #	
	Site #1-1	Site #2-2
Carbon	78.9	85.2
Hydrogen	5.4	5.1
Nitrogen	1.0	0.8
Sulphur	4.9	3.3
Oxygen	9.8	5.6
Atomic H/C	0.82	0.72
Yield ^a	0.28	0.36

^a: wt., tar refuse dry basis.

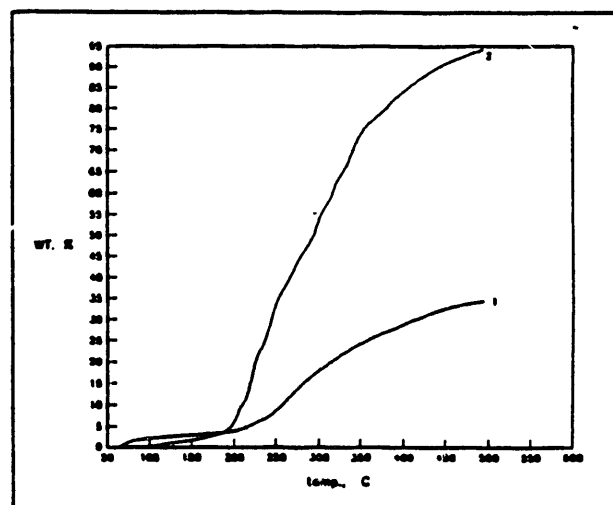


Figure 2.4. Simulated distillation of site #1-1 tar (gpc fractions).
 1 - mol. weight >400 (av. 1000)
 2 - mol. weight >400 (av. 330).

Table 2.7. Molecular weight (MW) distribution of tar by GPC.

MW range	Cumulative elution vol. ml	wt, % Sample #							
		1	2	3	4	5	6	7	8
Site #1 (grab) ^a									
>300	120	28	30	26	48	-	-	-	-
<300	240	72	70	74	52	-	-	-	-
Site #2 (grab) ^b									
>400	150	58	44	24	47	64	48	81	82
<400	250	44	56	76	53	36	52	19	18
Site #1 (bulk) ^c									
>400	100	34	-	-	-	-	-	-	-
<400	300	66	-	-	-	-	-	-	-
Site #2 (bulk) ^c									
>400	100	-	40	-	-	-	-	-	-
<400	300	-	60	-	-	-	-	-	-

^a: SX-4 column; mw exclusion >1400; void vol., ml-108; chloroform

^b: SX-3 column; mw exclusion >2000; void vol., ml-113; chloroform

^c: SX-8 column; mw exclusion >1500; void vol., ml-160; benzene

Table 2.8. Molecular weight distribution of THF solubles.

mw, range ^a	wt, % Bulk sample #	
	Site #1-1	Site #2-2
>400	57	64
<400	43	36

^a: conditions as for SX-8 column in Table 2.7.

Table 2.9. Simulated distillation of tar (qc).

Temperature range (°C)	Grab sample			Bulk sample	
	Site #1(1)	Site #1(3)	Site #1(4)	Site #1-1	Site #2-2
IBP - 250	29	21	7	25	13
250 - 300	20	18	10	14	9
300 - 350	15	15	10	14	12
350 - 400	8	11	12	8	11
400 - 450	4	5	9	6	8
450 - 500	3	3	9	3	8
Non-dist. residue ^a	21	27	43	30	39

a: The yields of non-distillable residue correspond roughly to yields of the GPC fraction with mw >300.

Table 2.10. Hydrocarbon type analysis^a.

gpc fraction	Cum. elution vol, ml ^e	Yield %	wt. % Hydrocarbon type			Loss
			Aromatics ^b	Polar ^c	Polar ^d	
Site #1-1						
50	22		1	30	48	21
250	78		61	21	9	9
Site #2-2						
50	27		4	28	32	36
250	73		45	34	10	11

a: chromatographic separation on alumina/silica gel column
 b: eluted with 15% benzene in pentane
 c: eluted with benzene
 d: eluted with 40% methanol in dichloromethane
 e: molecular weight decreases with increasing the elution volume.

weight below 300 - 400 varied from as much as 76 percent to as little as 18 percent of total tar.

The tar molecular weight distribution is reflected in the volatility of tar. The higher molecular weight (mw >400) portion of tar corresponds roughly to +500°C non-distillable residue. The aromatic hydrocarbons are mostly concentrated in the lower molecular weight (mw <400) portion of tar.

The major hydrocarbons identified in the soluble tar were naphthalenes, anthracenes, phenanthrenes and pyrenes. Appendix 2, Figure A2.3 presents an example of the spectra of the soluble tar and a detailed list of hydrocarbons identified in tar by capillary gas chromatographic-mass spectroscopy methods. As expected, the tar refuse contains a mixture of complex hydroaromatic compounds generated during coal carbonization.

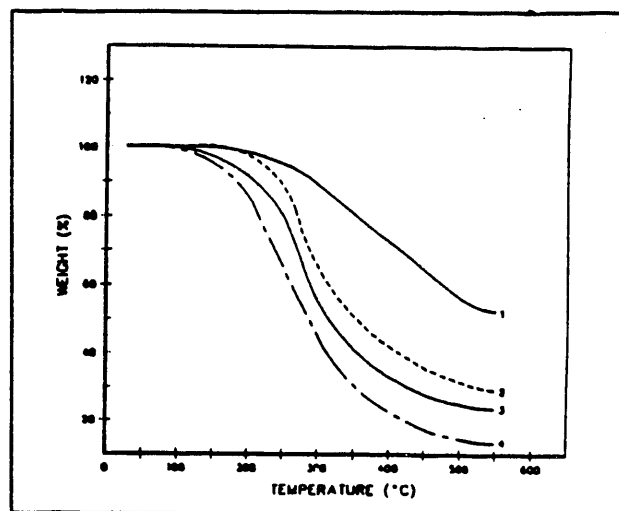


Figure 2.5. Thermogravimetric analysis of site #2-2 tar (gpc fractions).

1 - cum. elut. vol., ml 50
 2 - cum. elut. vol., ml 75
 3 - cum. elut. vol., ml 100
 4 - cum. elut. vol., ml 300

} mw >400 (av 1100)
 mw <400 (av 350)

To estimate the concentrations of target compounds in the contaminated samples as well as to evaluate the efficiency of the clean-up process in their removal, Site #1-1 and Site #2-2 feeds and process products were analyzed by Radian Corp. Selected results are shown in Tables 2.11 and 2.12. Appendix 2 presents the data as received.

2.2.2. Agfloat Process

The Agfloat process was developed for beneficiation of coals. The procedure is a combination of agglomeration and floatation methods. Mixing a coal/water/bridging liquid slurry in a stirred tank of solids concentration of 25-40% at about 2000 rpm produces microagglomerates, which are then separated from the dispersion by floatation. The application of this procedure to the clean-up of solids contaminated with tarry and/or oily residue is based on the presumption that these contaminants will play the role of bridging liquids which are required for coal to agglomerate (Figure 2.10).

The nature of the tar refuse samples to be cleaned by the Agfloat process necessitated some modification of the standard procedure. Firstly, to accommodate soil samples that contain a high proportion of coarse solids, a stirred tank was replaced by a tumbler.

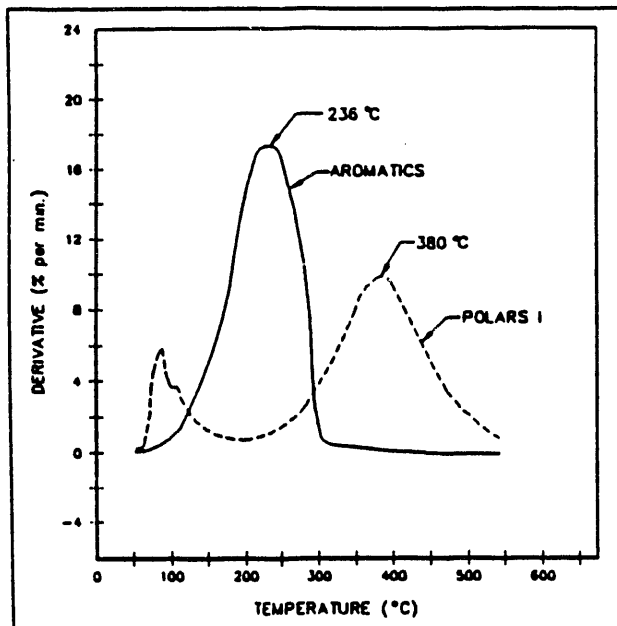


Figure 2.6. Thermogravimetric analysis of site #1-1 hydrocarbons (mw<400).

Table 2.11. Site #1-1 radian analysis.

Sample	µg/g Volatiles	Semivolatiles	Total
Feed	74.06	2,567.00	2,641.06
Froth	18.45	7,139.00	7,157.45
Clean Soil ^a	2.20	197.26	199.46
Water ^b	826.36	3,320.00	4,146.36

a: 0.17 % tar; b: µg/L.

Secondly, to facilitate an optimal transfer of viscous tar from mineral matter onto coal, the process was operated at elevated temperatures. Figure 2.11 shows the effect of the tumbling temperature on the amount of tar retained in clean soils. The amount retained decreased rapidly as the temperature approached 70°C, and then levelled off. Based on these results all subsequent experiments were performed at temperatures between 74 and 80°C. The experiments and product analyses were conducted as described in Section 2.1.1.

Results with Site #1 (Grab sample)

Table 2.13 presents the clean-up data for samples 1 to 4. The key features depicted are:

- The clean-up objective was to retain no more than 1000 parts per million (0.1%) of soluble tar in the tailings. Only sample #2 achieved this target level.

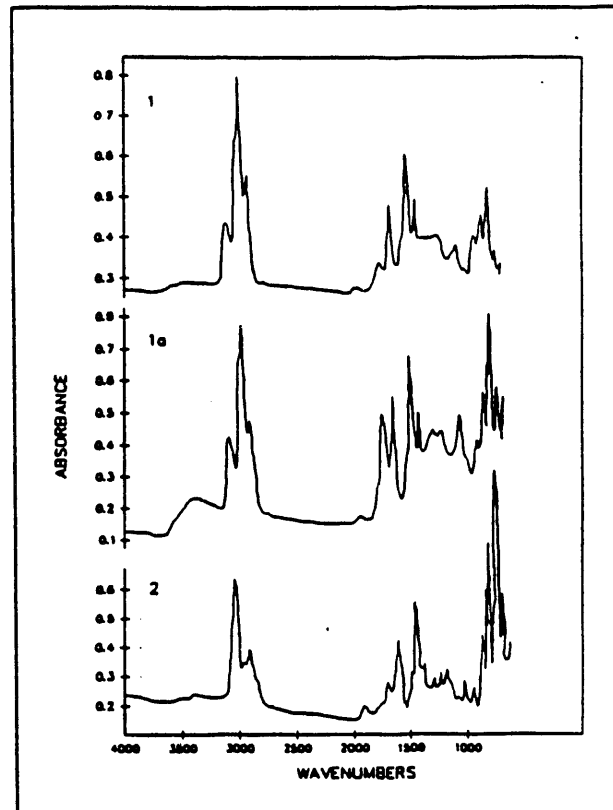


Figure 2.7. Infrared spectra of site #1-1 tar (gpc fractions)

1 - cum. elut. vol., ml 50
 1a - cum. elut. vol., ml 100 } mw > 400
 2 - cum. elut. vol., ml 300 } mw < 400

Table 2.12. Concentration of polynuclear aromatic hydrocarbons (ppm).

Hydrocarbon	Site #1-1		Site #2-2	
	Feed	Clean soil ^a	Feed	Clean soil ^b
Naphthalene	110	2	310	7
2-Methyl-naphthalene	270	5	720	-
Acenaphthylene	110	6	360	6
Fluorene	57	3	280	8
Fluoranthene	76	18	410	12
Phenanthrene	210	42	1200	68
Chrysene	38	9	250	10
Pyrene	110	28	50	14

a: 0.17% tar; b: 0.25% tar.

- The tar retention in the tailings of sample #1 was high (0.8%). It is likely that there is a relation between that retention of tar and the presence of coke-like inclusions in the soil, since sample #1

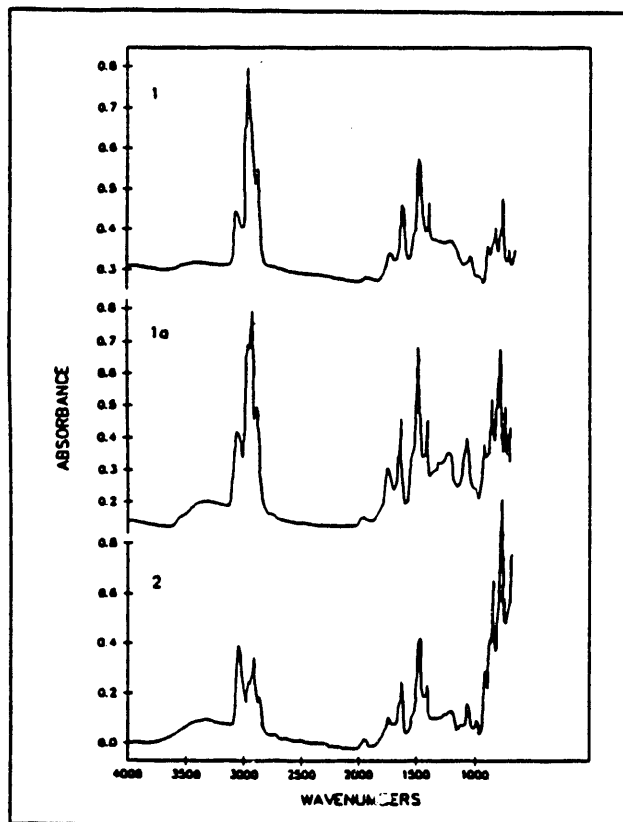


Figure 2.8. Infrared spectra of site #2-2 tar (gpc fractions).
 1 - cum. elut. vol., ml 50
 1a - cum. elut. vol., ml 100 mw >400
 2 - cum. elut. vol., ml 300 mw <400

Table 2.13. Site #1 clean-up of tar refuse grab samples by Aglofloat procedure.

Sample #	1	2	3	4
Process Parameters				
Solids concentration, % ^a	37	42	42	22
Tar/coal (daf) ratio	0.44	0.07	0.33	0.12
Tar Refuse-feed				
% Tar (toluene soluble, ml)	8.57	1.20	5.44	2.60
% Combustibles (toluene soluble, ml)	30.3	2.0	7.7	14.5
Products				
Froth				
Yield, % of coal	140	104	137	50 ^b
% Ash	10.1	-	-	-
Tailings^c				
% Tar (ml)	0.8	0.0	0.3	-

a: Charge: sample as received, 200 g; coal, 35 g;

b: Floatation; slow, wet and not complete;

c: Solids with particle size below 3.3 mm.

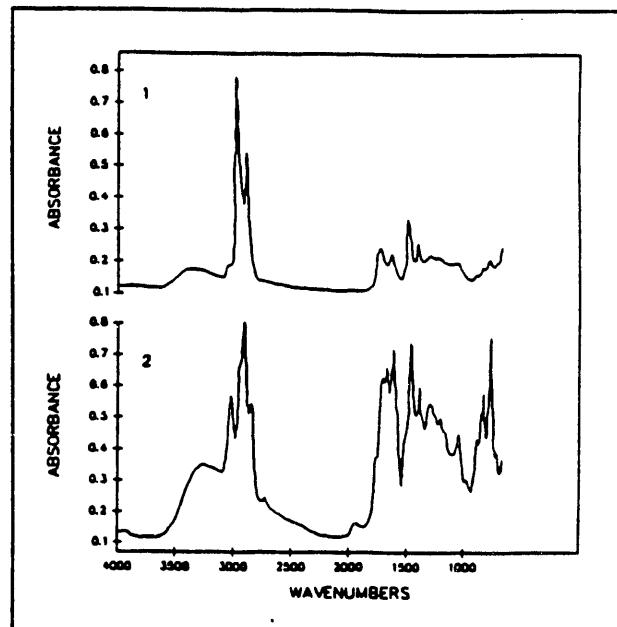


Figure 2.9. Infrared spectra of site #1-1 thf solubles (gpc fractions). 1 - mw >400; 2 - mw <400.

contained a high concentration of insoluble combustibles (30.3%).

- Sample 4 exhibited difficulties with formation of froth due to a large content of clay in the sample.
- Blending sample 4 with sample 2 resulted in better froth recovery.

Table 2.14 depicts the results of additional batch experiments conducted to improve the floatation characteristics of the blended samples. The key features presented are:

- By addition of a combination of frothers and collectors, better froth and combustibles recovery was achieved and almost all added coal and soluble tar was recovered in froth.
- The content of soluble tar in the tailings remained, in general, at 0.2 percent.
- The ash content of the froth varied from 14.3 to 33.9 percent, indicating mineral matter inclusion in all instances.
- Floatation carried out at room temperatures up to 25°C was faster and yielded drier froth with lower ash content than hot floatation (70-80°C).

Results with Site #2 (Grab sample)

On the basis of their composition, the samples from Site #2 can be divided into 2 groups (Table 2.15). Samples 2-4 in Group I have a very high tar concentration and very little mineral matter. The

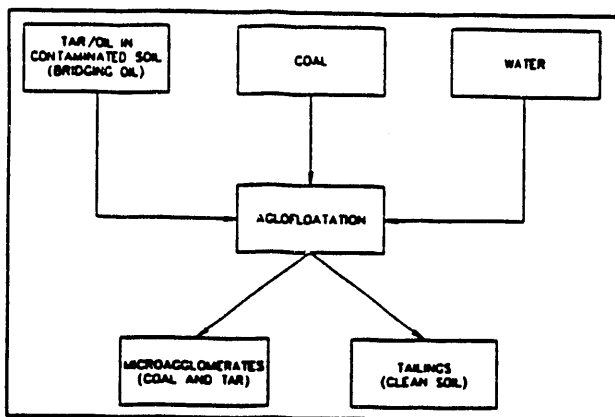


Figure 2.10. Principle of agglomeration process as applied to soil clean-up.

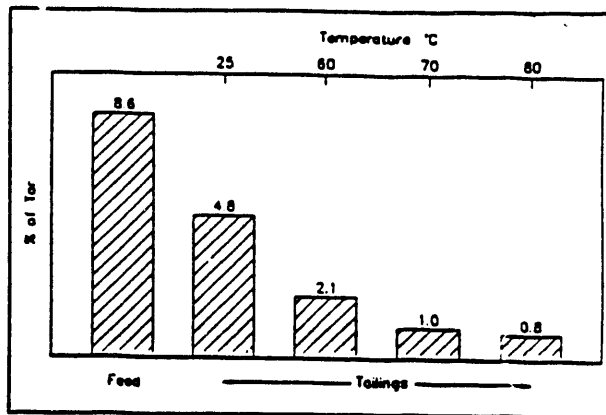


Figure 2.11. Effect of temperature on tar in clean soils.

Table 2.14. Co-processing of mixed tar refuse samples #2 and #4 from site #1.

	Hot floatation (70-80°)			Cold floatation (20-25° C)				
	1	2	3	Experiment #		6	7	8
Froth, gm	45.2	45.2	43.8	37.6	28.2	31.0	30.2	34.3
Tailings, gm	148.0	166.2	163.6	172.5	178.2	179.1	168.0	177.3
% Material loss, (m.f.)	7.2	0.8	2.2	1.3	2.6	1.3	5.5	0.7
% Tar in tailings	0.2	0.2	0.2	0.2	0.4	0.4	0.4	0.2
% Ash in froth	26.3	30.6	-	33.9	16.8	17.2	14.3	15.3
Additives								
Frother, A65, 1 ml	+	+	-	-	-	-	+	+
Silicate, 0.7 gm	-	+	-	+	-	-	-	-
Kerosene, 0.4 gm	-	-	+	+	-	+	-	+

Charge: sample 2, 150 g; Sample 4, 100 g; coal, 35 g; Solids concentration - tumbling, 38%; floatation, 10%; % tar in feed, 1.5.

samples in Group II are characterized by low to medium tar content and clay-like solids.

Table 2.16 shows the results of the Aglofloat tests for samples 1 to 8. The following are the main conclusions:

- The samples in Group I agglomerated readily resulting in large and well developed agglomerates and low concentration of tar in tailings.
- The samples in Group II responded poorly to the Aglofloat processes, especially to floatation, which

Table 2.15. Site #2 average composition.

	Group I Samples 2-4 (%)	Group II Samples 1, 5-8 (%)
Soluble Tar	38 - 60	<1 - 5
Other Combustibles	14 - 31	5 - 28
Mineral Matter	3 - 13	51 - 87

in all cases but one (sample 8) was very slow and wet. Samples 5 and 6 had a poor froth recovery. In samples 7 and 8, a part of the mineral matter floated together with coal yielding froth with a very high ash content.

In order to improve the floatation characteristics of samples 1 and 5, a series of batch experiments was conducted, in which prior to processing, these samples were blended with Group I samples. The results are shown in Tables 2.17 and 2.18. The key results were:

- Better froth recovery was achieved for blended samples.
- Ash content in the froth was much lower (8.2 to 9.9 percent) than in previous tests.
- The percentage of tar retained in the soil was still high.

Table 2.16. Site #2 clean-up of tar refuse samples by Aglofloat procedure.

Sample #	Group I				Group II			
	2	3	4	1	5	6	7	8
Charge, gm								
Coal	140	140	140	30	40	20	20	20
Tar Refuse	51	48	26	200	200	100	100	100
Solids Concentration, % in water	34	37	34	39	37	27	26	26
Tar/coal (daf) ratio	0.16	0.23	0.09	0.15	0.03	0.03	0.30	0.03
Tar Refuse - feed								
% Tar (mf)	66.9	67.6	48.7	2.2	0.6	0.6	5.9	0.7
% Mineral matter (mf)	8.4	3.3	15.2	66.5	64.4	94.1	68.4	81.0
Products								
Froth								
Yield, % of coal	116 ^a	124 ^a	95 ^a	101 ^c	39 ^{c,d}	84 ^{c,d}	350 ^e	149 ^e
Tailings								
% Tar (mf)	0.1	0.1	28.9 ^b	-	-	0.5	1.1	0.1

a: dry agglomerates; b: not dispersed lumps of tar retained in tailings; c: slow, wet flotation; d: flotation not complete; e: high ash content.

Table 2.17. Co-processing of tar refuse samples 1 and 3 by Aglofloat procedure^a.

	Experiment #							
	1	2	3	4	5	6	7	8
Charge, gm								
Coal	50	50	75	75	75	75	75	75
Sample #1	80	80	80	80	100	200	200	200
Sample #3	20	21	21	19	20	20	21	20
Tar/coal %	27	29	19	17	18	21	22	21
Solids Concent., %	28	28	31	31	28	36	36	36
Products, gm								
Froth ^b	65.0	77.8	87.8	83.5	101.5	118.7	104.6	124.2
Tailings	61.6	55.4	64.6	62.0	78.7	148.3	163.5	141.4
% Material loss, (m.f.)	8.4	4.2	7.0	4.0	0.3	1.0	0.7	1.3
% Tar in feed (m.f.)	15.2	15.7	15.7	15.0	13.3	8.1	8.1	8.1
% Tar retained in Tailings	1.3	0.4	1.8	0.6	0.6	0.7	1.2	0.4
Froth - % ash	-	8.9	-	8.6	8.2	9.2	-	-

a: variables investigated: 1. tumbling (exp. 1 & 3) vs. mixing in a stirred tank at 1900 rpm (exp. 2 & 4). 2. tar/coal ratio (exp. 2 & 4). 3. solids concentration (exp. 5 & 6). 4. pH=4 (exp. 7) vs. pH=9 (exp. 8)
b: fast and dry flotation.

Table 2.18. Site #2 co-processing of group I and II samples.

	Experiments ^a				
	1	2 ^b	3 ^b	4	5
Group I					
Sample #3	X	X			
Sample #4			X		
Blend #3 & #4				X	X
Group II					
Sample #1	X			X	
Sample #5		X	X		X
% Tar (mf)					
Feed	15.7	7.0	7.5	9.4	8.7
Tailings	0.4	0.5	6.5 ^c	1.5	1.1
% Ash - Froth	8.7	-	-	9.8	9.9

a: Tar/coal ratio: 0.16 - 0.28; Solids concentration: 40%; b: Slow flotation; c: Not dispersed lumps.

Results with Site #1 and Site #2 (Bulk Samples)

During the evaluation of the bulk samples from Site #1-1 and Site #2-2, it appeared that coal coated with tar did not float properly. It simply hindered the product separation (Table 2.19). Poor froth recovery in the tests was coincidental with large quantities of coal and high tar retention in tailings. The experiments, in which coal used in a clean-up procedure and tar extracted from Site #2-2 for tar refuse was subjected to Aglofloat procedures, indicated that this particular tar suppresses froth formation (Table 2.20).

Table 2.19. Bulk samples clean-up by Aglofloat procedure - froth recovery.

	Froth, g		received	Tailings % Tar
	max ^a	min ^b		
Site #1-1	78	45	42	2.9
Site #2-2	138	68	57	4.0

a: based on a combined amount of coal and combustibles present in tar refuse solids; b: based on an amount of coal.

Table 2.20. Floatation of coal used in a clean-up of tar refuse (wt % of coal feed)^a.

Products	Collector		Tars Frother ^d
	Kerosene ^b	Kerosene ^b Frother ^d	
Froth	78.9	91.3	45.0
Tailings	18.8	8.4	55.0

a: Coal, 100g; b: 0.5 ml; c: 5.0 g; d: 1 ml Aero 65.

Effect of Some Process Variables

To improve the response of bulk tar refuse samples to the clean-up process, the following variables were studied:

- slurry pH using H₂SO₄ or NaOH
- addition of froth collector kerosene, frother, diesel, etc.
- conditioning of refuse xylene, benzene, dichloromethane
- coal properties bituminous or sub-bituminous type
- agitation mode and intensity

The results of the tests are discussed below.

Slurry pH: Changing the pH of slurry did not have any beneficial effect on floatation (Table 2.21).

Froth Collector: Addition of a froth collector improved the froth recovery significantly (Tables 2.22 and 2.23). Kerosene, diesel and coal distillate V-138 have about the same froth collecting properties. With the increasing addition of a collector, the froth recovery increases, and the amount of tar retained in tailings decreases. In general, the tar content of clean soil is much lower than that obtained in the absence of a froth collector (Table 2.19) but it still exceeds 1 percent.

Slurry Conditioning: A small quantity of solvent (conditioner) was added during tar refuse/coal slurry

Table 2.21. Site #2-2 (bulk) clean-up by Aglofloat procedure - effect of pH on froth recovery.

	Experiment #				
	1	2	3	4	5
pH		8.4	9.7	10.6	3.9
Froth ^a received (g)	58.5	65.8	62.7	18.1	52.2

a: max., expected: 138 (g); min., expected: 68 (g).

Table 2.22. Site #2-2 (bulk) clean-up by Aglofloat procedure - effect of froth collector.

	Experiment #			
	1	2	3	4
Collector	Kerosene	Diesel	Diesel	V-138
ml/75g coal	0.5	0.5	4.5	1.0
Froth Received, g	103.6	118.0	133.0	115.0
% Tar in Tailings	2.0	1.6	1.0	1.9
% Ash in Froth	8.2	11.8	12.8	-

Table 2.23. Bulk samples cleanup by Aglofloat procedure - effect of froth collector^a.

	Site #1-1	Site #2-2
Charge, g		
Tar Refuse	200	200
Coal	50	75
Water	300	300
Froth received, g	75	118
% Tar in Tailings	1.25	1.62

^a Diesel, 1% of coal (daf).

Table 2.24. Site #2-2 (bulk) clean-up by Aglofloat procedure - effect of conditioning.

	Experiment # ^a		
	1	2	3
Conditioner	Xylene ^a	Benzene ^b	Dichloromethane ^b
% Tar in Tailings	1.4	1.9	1.3

a: 2 ml/200g sample added at mixing stage; b: 5 ml/200g sample added 12 hrs prior to mixing; c: Diesel as froth collector.

preparation to enhance the release of tar from soil. Table 2.24 indicates that no significant change in the amount of tar remaining in the soil was observed after the addition of the conditioners.

Effect of Coal Properties: Application of different coals in the agglomeration step did not significantly change the amount of froth collected or the amount of soluble tar retained in the soil (Table 2.25). The results indicated that, with proper optimization, a

Table 2.25. Site #2-2 (bulk) cleanup by Aglofloat procedure using various coals^a.

	Experiment #						
	16 ^b	17 ^b	18	19	20	21	22
Coal	PSE&G	Upper Freeport	PSE&G	PSE&G	Kemmerer ^c	Ohio	Upper Freeport
Froth received, g	102.4	95.0	120.0	115.2	117.0	114.5	130.1
% Tar in Tailings	3.0	3.6	1.6	2.5	2.1	1.8	1.5

a: mixing at 2400 rpm; froth collector: diesel - 1 ml/75 g coal; b: no froth collector added; c: subbituminous.

number of different coals may be used with the process.

Effect of Agitation: One of the Site #2-2 batches contained visible oily lumps that were not dispersed during tumbling. In this case, two agitation methods, stirring and tumbling, were tested for the Aglofloat process (Table 2.26). The results suggest that increased agitation in the stirred tank increased the amount of froth received and decreased the amount of tar remaining in the tailings.

In a large scale operation and when there are oversized solids in contaminated samples, agitation in the stirred tank may not be feasible.

During tumbling, an increase in agitation can be accomplished by increasing the solids concentration. The higher the solids concentration the better the interaction between coal and tar refuse, and the attrition between solid particles. In consequence, a better transfer and absorption of tar onto coal should be achieved.

Table 2.26. Site #2-2 (bulk) clean-up by Aglofloat procedure - effect of agitation.

	Experiment ^a	
	Tumbling	Stirring ^b
Temp °C	77	84
Froth received, g	99.1	103.6
% Tar in Tailings	4.6	1.4

a: diesel as froth collector added; b: at 2400 rpm.

Table 2.27. Clean-up of tar refuse samples.

Source	Tar retained in soil (-3mm particle size)		
	Aglofloat standard ^a	Aglofloat optimized ^b	Clean Soil process ^{c,d}
Site #1-1	2.9	1.26 ^a	0.64
Site #2-2	4.6	1.62 ^b	1.60

a: Mixing in a tumbler (90 rpm); b: Mixing in a stirred tank, 2400 rpm; c: Diesel as froth collector added: 1-2% of coal; d: Solids concentration in a tumbler increased to 65-75%.

The following section discusses development of the Clean Soil process based on the above process concept. This process uses solids loading in the tumbler of between 60 to 85 percent, in contrast to the Aglofloat process which uses solids loading of 30 to 35 percent.

2.2.3. Modification of Aglofloat Process - Development of Clean Soil Process

The major difference between the Aglofloat and the Clean Soil processes is that solids loading with the Clean Soil process is about twice as high as with the Aglofloat process. The high solids concentration results in increased abrasive action between solid particles of coal and tar refuse, more attrition of the oily lumps and, hence, better removal of soluble tar from the soil.

Table 2.27 compares test results for standard and optimized Aglofloat procedures, and unoptimized Clean Soil procedures. Samples from Sites #1-1 and #2-2 were selected because of poor results in the Aglofloat process (Section 2.2.2.). They provided a very demanding test for the Clean Soil process. The results show significant improvements for sample #1-1 in the Clean Soil process, and none for sample #2-2. Table 2.28 shows that the ash values for solids in sample #2-2 are significantly lower than expected for pure mineral matter. This indicates that the sample contained coke and char.

Thus a lack of improvement in the case of the Site #2-2 sample can be directly related to the presence of tar saturated coke and chars. These materials

Table 2.28. Particle size distribution and ash concentration of tar refuse toluene insoluble solids in tar contaminated soils.

Particle size, mm	Site #1-1		Site #2-2	
	wt %	Ash, %	wt %	Ash, %
0 - 1.0	73	91.5	42	66.9
1.0 - 3.3	14	91.9	15	70.8
>3.3	13	78.2	43	82.7

Table 2.29. Clean Soil process – effect of froth collector on tar removal.

Froth Collector ^a % of coal (daf)	% Tar retained in soil (-3 mm particle size)	
	Site #1-1	Site #2-2
2	0.64	1.60
5	-	0.55
8	0.16	0.17

a: Bitumen/Diesel (1:1)
 Charge: Site #1-1: sample/coal/water (g) - 530/140/160
 Site #2-2: sample/coal/water (g) - 350/250/200

have a high affinity for tar, and therefore are not likely to release it during processing.

In order to effectively reduce tar retention in clean soils, the combustibles must be removed as well. One feasible way of achieving this is by floatation. The experimental results in which coke floatation was increased by the addition of froth collector during the separation step in the Clean Soil process, are presented in Tables 2.29 and 2.30. When the oil addition was increased, the tar content decreased in the processed soil. This was also accompanied by a reduction of combustibles. It should be mentioned that at 8 percent oil addition there was no need for frother (Aero 65 or MIBC) which was used otherwise.

It was noted that the residual tar was not evenly distributed in the clean soil obtained from sample #1-1 using this process. The coarse fraction retained more tar (Table 2.31). Since tar is associated with coke, the presence of tar-coated coarse coke particles implies that grinding may be a necessary step to ensure adequate tar removal by floatation. Various alternate process schemes which were undertaken in order to reduce the tar content in the processed soil, are listed in Table 2.32.

For contaminated soils such as those from Site #1-1 and Site #2-2 which are characterized by:

- the presence of tar that suppresses coal floatation, and
- the presence of coke, chars and slag of various size.

Grinding of solids to a particle size suitable for floatation and selective oil addition to assist floatability of carbonaceous materials are both recommended. Based on this recommendation, a procedure for processing tar/oil contaminated soil has been developed. The procedure is outlined in Figures 2.12.

The use of both froth collector at the floatation stage during processing and reprocessing may be

Table 2.30. Clean Soil process – char/coke removal^a.

Feed	% Tar-free combustibles in soil (-3 mm particle size)	
	Feed	Clean Soil
Site #1-1	12.0	1.6
Site #2-2	30.2	8.7

a: at 8% froth collector addition.

Table 2.31. Site #1-1: Tar content in various size fractions.

Fraction size mm ^a	Fraction yield %	Tar content of fraction %
0 - 0.6	60.5	0.34
0.6 - 1.0	20.6	0.61
1.0 - 3.3	18.8	1.59

a: tar content in 0 - 3 mm soil, 0.64%.

Table 2.32. Additional Clean Soil process schemes tested and their results.

Procedure	Processed refuse particle size mm	Fraction recovery as clean soil %	% Tar	
			Before treatment	After treatment
Selective Oil addition ^a	0-3.3	-	4.3-8.9	0.17
Refloatation	0-1.0	59	0.51	0.27
Refloatation with coal	0-1.0	94	0.51	0.17
Float-sink	0-3.3	95	0.85	0.40
Grinding & Reprocessing	0-3.3	93	0.85	0.26

a: in the first treatment.

required depending on the nature of a sample. Selective grinding can also be used during the mixing stage introducing balls and rods which intensifies the friction in a tumbler. The amount of reject depends on the severity of grinding. During separation by floatation, various amounts of middlings (solids suspended in water) may be produced as an end product. Depending on the ash and tar/oil contents, the middlings can be combined either with clean soil or froth.

Figures 2.13 to 2.15 and Tables 2.33 to 2.36 describe in detail the processing of samples from Site #1-1 (see also Tables A2.1 to A2.5 in Appendix 2). The results obtained from the first stage of the process were:

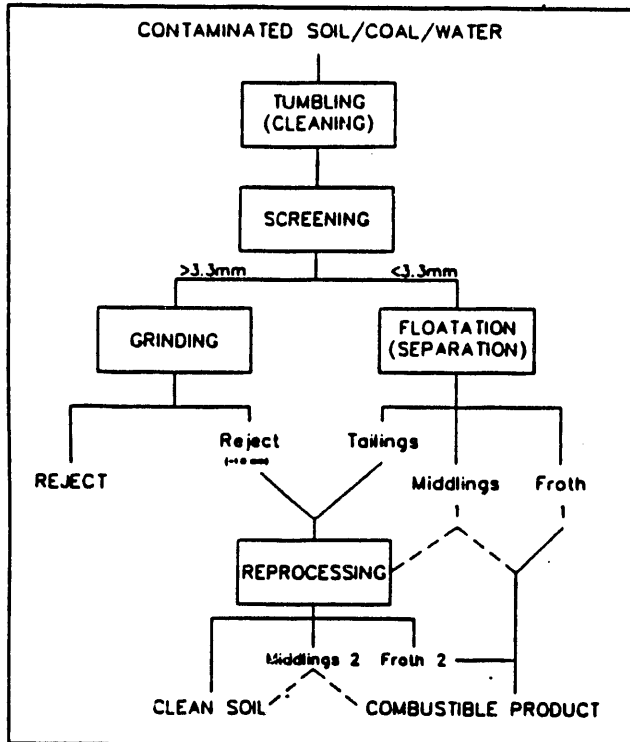


Figure 2.12. Process schematics for cleaning tar/oil contaminated soils.

Table 2.33. Site #1-1: Clean-up of contaminated soil process conditions.

Tumbling	
1. solids concentration	65-75%
2. tar to coal (daf) ratio	0.18
3. temperature	782°C
4. rotation speed	60 rpm
5. residence time	12 min
6. metal balls	2650 g

Grinding	
7. crushing with a hammer followed by	
8. wet grinding in a ball mill, 20 min	

Floatation	
9. froth collector: Bitumen/diesel blend (1:1)	
10. froth collector to coal (daf) ratio, 0.10	
11. conditioning: 1 min at 2400 rpm	
12. frothing: temp 35-45°C, 1100 rpm; solids concentration, 20-25%	

Reprocessing	
13. tumbling: coal and water added; otherwise as in 3 to 6	
14. floatation: froth collector (9) added; collector to coal (daf) ratio, 0.04; otherwise as in 11, 12	

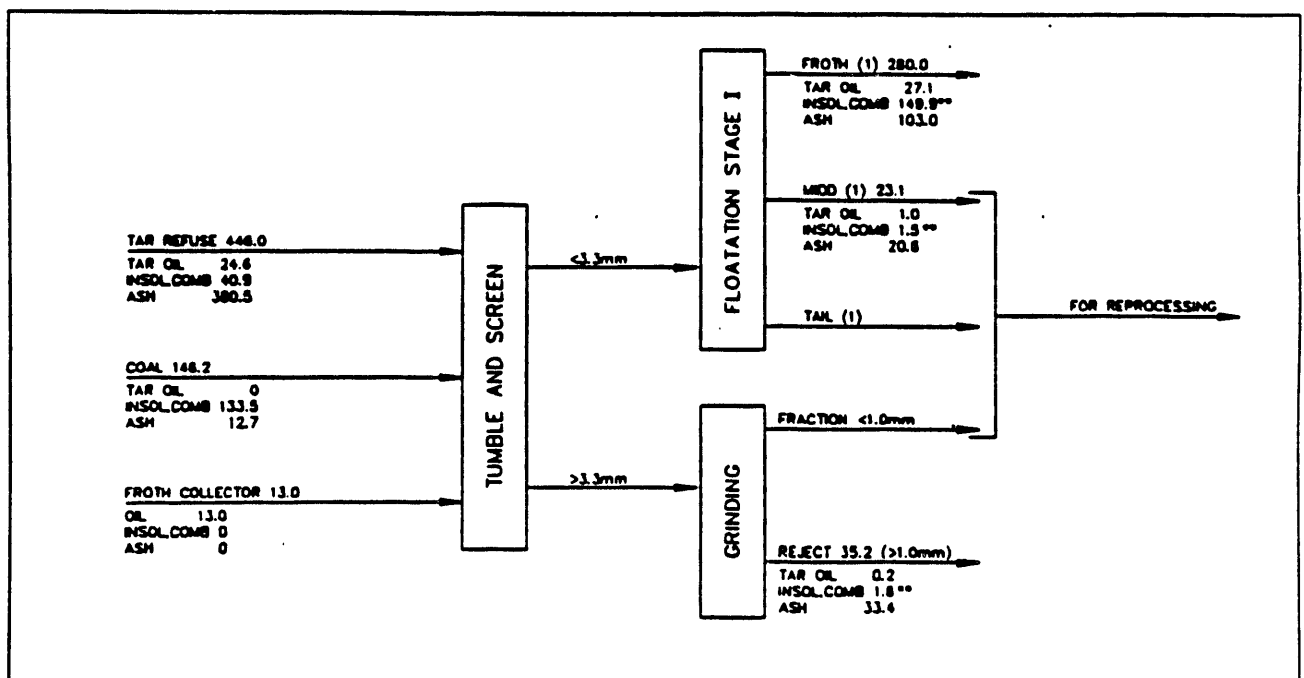


Figure 2.13. Material balance for clean soil process. (Site #1-1; units in grams)
*from forced balance on reprocessing step; ** by difference.

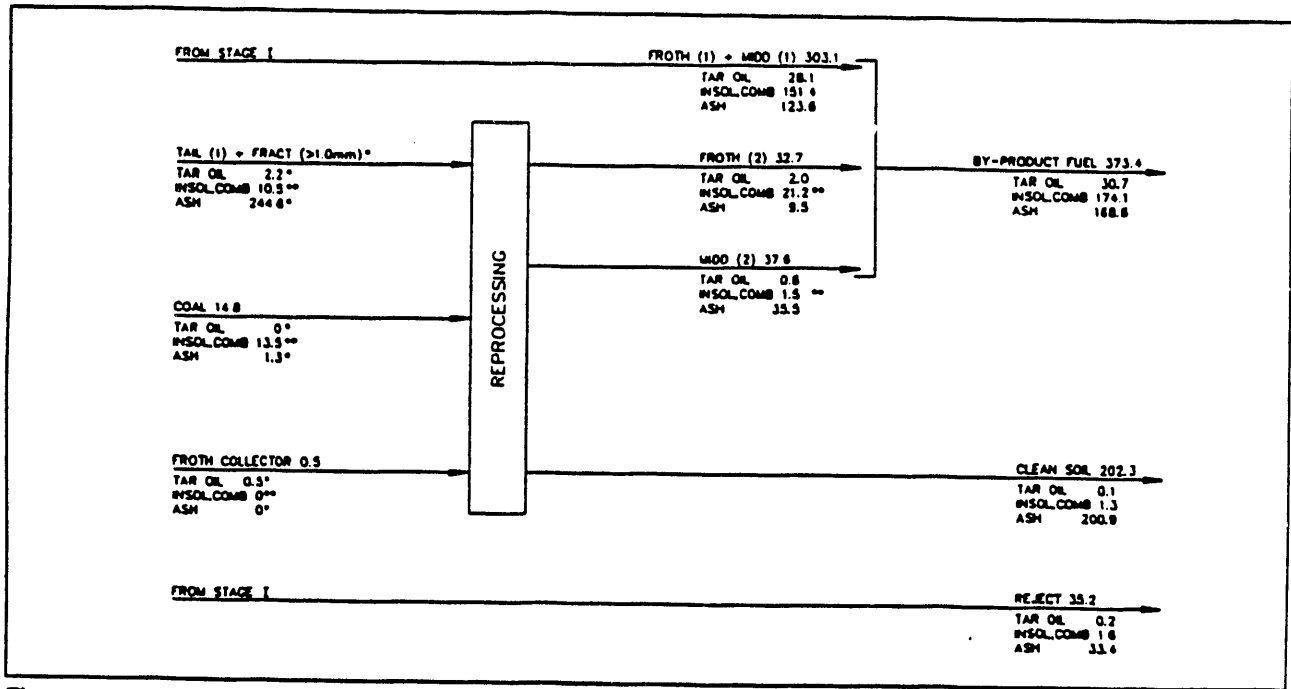


Figure 2.14. Material balance for Clean Soil process. (Site #1-1, units in grams)
*from forced balance on reprocessing step; ** by difference.

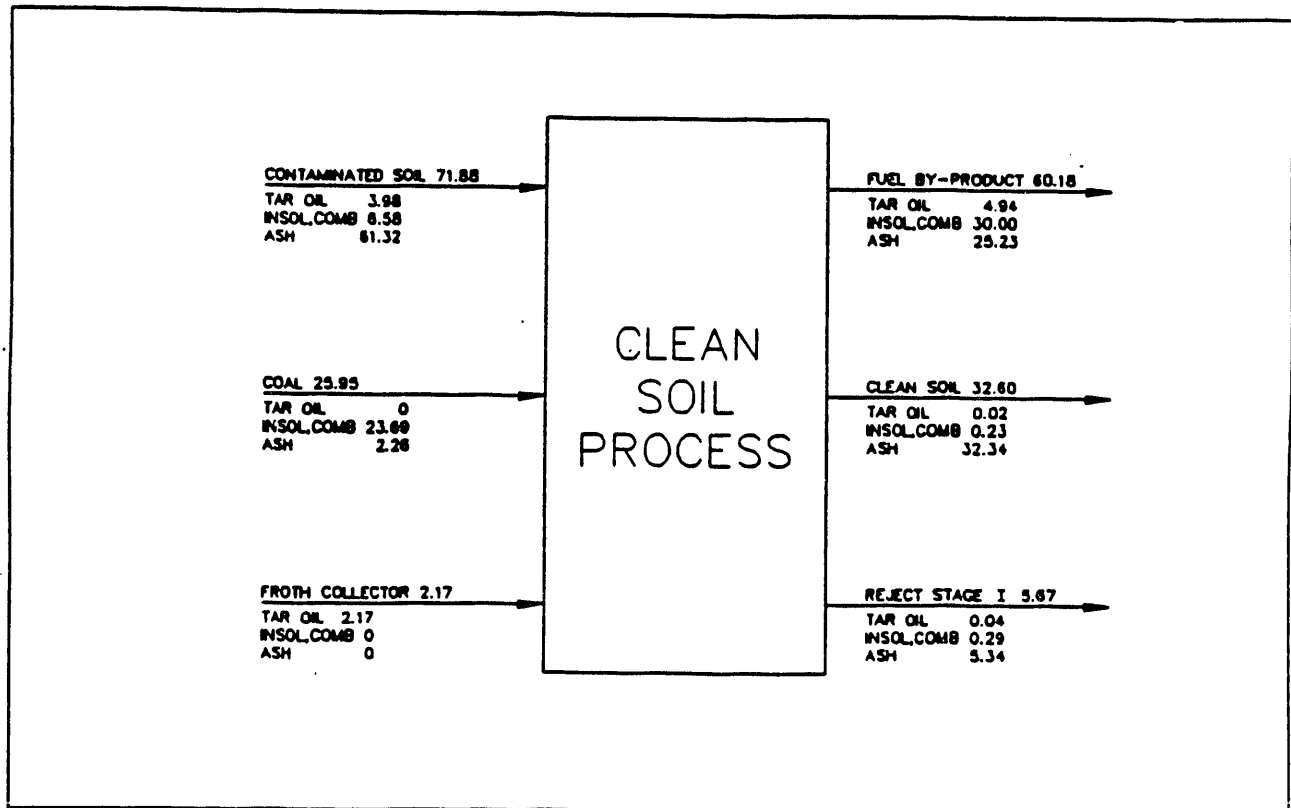


Figure 2.15. Overall material balance for Clean Soil process. (Site #1-1, Feed=100)

Table 2.34. Contributions of different streams to fuel product.

Stream	% Contribute	wt. g.	Tar oil		Insol. comb.		Ash	
			g	%	g	%	g	%
FROTH 1	(75%)	280.0	27.08	9.67	149.88		103.04	36.8
MIDD 1	(6.2%)	23.1	1.01	4.37	1.53		20.56	89.0
FROTH 2	(8.8%)	32.7	2.03	6.20	21.19		9.48	29.0
MIDD 2	(10.1%)	37.6	0.61	1.61	1.53		35.46	94.3
COMBINED	(100%)	373.4	30.73	8.2	174.13	46.7	168.54	45.1

Table 2.35. Properties of clean soil and reject combined.

Component	Clean Soil		Reject Stage I		Clean soil and Reject combined	
	g	% ^a	g	% ^a	g	% ^a
Tar Oil	0.14	0.07	0.24	0.68	0.38	0.16
Insol. Comb.	1.28	0.63	1.59	4.51	2.87	1.21
Ash	200.88	99.30	33.40	94.81	234.28	98.63

^a percent of product stream

- 74.9 percent of tar oil including froth collector and 86.8 percent of insoluble combustibles (coal and coke) were recovered in the Froth (1) and MIDD (1) streams (Figure 2.13).
- The Tail (1) and <1.0 mm ground refuse fraction, which contained most of the soil required further reprocessing to meet the target of 0.1 percent or less of tar oil in clean soil.

In the second processing stage (Figure 2.14.) TAIL (1) and <1.0 mm ground refuse fraction were reprocessed with the addition of coal and froth collector. The product fuel (FROTH (2) and MIDD(2)) were then combined with the fuel obtained in stage one of the process. The second stage process results were:

- An additional 6.8 percent of tar oil was recovered in the Froth (2) and MIDD (2) streams.
- The amount of the retained tarry contaminant in the clean soil, which represents close to 50 percent of the contaminated solids, is 0.07 percent (Table 2.35).
- The total recovery of tar oil and froth collector stages 1 and 2 was 81.7 percent. This loss of 18.3 percent of organics is attributed to volatility of tar and froth collector and could be prevented by recondensation.

Different contaminated soil to coal ratios were tested (Tables 2.37). The tests showed that increasing the contaminant soil to coal ratio results in higher

tar retention in clean soil. Presumably, this is due to the fact that less surface area of coal is available for tar adsorption.

The tests with different types of contaminated soils (Table 2.38) showed that a variety of tar refuse samples can be treated with the Clean Soil process provided that, for some samples, a suitable froth collector to aid floatation and selective grinding, and to release the tar occluded in pores of coke, char and slag is applied. Table 2.38 also shows that oil spills can be cleaned without additional grinding or reprocessing.

2.3. Summary and Conclusions

A process for clean-up of soil contaminated with oily/tarry materials has been developed, i.e. process is based on application of coal for contaminants adsorption.

Twelve small grab samples and five bulk samples from two tar refuse contaminated sites were tested in the project. The samples had tar content ranging from 1.1 to 5.9 percent, insoluble combustibles content ranging from 1.9 to 30.9 percent, and mineral matter content of 2.9 to 92.2 percent.

The batch tests using the Aglofloat process showed that only a few samples produce clean soil with soluble tar content of less than 0.1 percent.

Table 2.36. Distribution and recovery of feed components in products and by-products.

Component	Feed		Fuel product		Clean Soil		Reject Stage I		Component Recovery %*
	g	(%)*	g	%	g	%	g	%	
Tar Oil	38.1	(100)	30.73	(80.7)	0.14	(0.4)	0.24	(0.6)	81.7
Insol. Comb.	187.9	(100)	174.13	(92.7)	1.28	(0.7)	1.59	(0.8)	94.2
Ash	394.5	(100)	168.54	(42.7)	200.88	(50.9)	33.40	(8.5)	102.1

* Percent of component feed.

Table 2.37. Site #1-1: Clean-up of contaminated soil - Effect of tar to coal ratio on clean-up.

Tar/Coal (daf) ratio	% Tar in clean soil
0.18 ^a	0.04 - 0.14
0.27	0.15
0.39	0.23

a: eg. 280 coal units/1000 soil units.

Treatment of some samples also resulted in poor coal recovery in the floatation step; however, improved insoluble combustibles recovery was achieved for these samples through addition of froth collectors.

Experiments using mixed tar refuse samples showed that better froth recovery and lower ash content in the froth can be achieved. Blending of samples did not, however, reduce the soluble tar content in clean soil.

Studies of the effect of process variables on combustibles recovery and soil clean-up showed that slurry pH, slurry conditioning and coal properties did not have any effect on the process results. In contrast, addition of froth collectors, increased slurry temperature, tumbling and agitation in the agglomeration step, all resulted in increased insoluble combustibles recovery and decreased tar oil content in clean soil.

Two major limitations were encountered in the use of the Aglofloat process, namely:

- inability to process solids with particle size over the floatability limit, and
- inability to clean extractable organic materials trapped in porous solids (slags, coke, char).

These limitations were addressed and overcome by development of the Clean Soil process.

Table 2.38. Clean-up of contaminated soil - various samples.

Type of contaminant	Feed	Contaminant concentration, wt % Processed soil		
		Aglofloat	Clean soil process	Grinding and reprocessing
Tar #1(1)	8.6	0.83	-	0.07
Tar #1(2)	1.2	0.00	-	-
Tar #1(3)	5.4	0.29	-	-
Tar #1(4)	1.6	0.20	-	-
Tar #2(2)	66.9	0.10	-	-
Tar #2(8)	0.7	0.10	-	-
Tar #1-1*	5.8	1.26	0.16	0.07
Tar #2-2*	10.6	1.62	0.17	-
Heavy Oil	9.7	0.25	-	-
Oil (light) Spills	2.0	-	0.17	-
Gasoline Spills	3.1	-	0.06	-
Diesel Spills	30.7	-	0.25	-
Oil (heavy) Spills I	43.0	-	0.08	-
Oil (heavy) Spills II	0.2	-	0.00	-

* froth collector required.

The Clean Soil process includes two stages: Stage 1 which is based on the Aglofloat process except that it includes higher solids loading in the tumbling step; and Stage 2 in which <1.0 mm reject and tailings from Stage 1 are ground and reprocessed. Tests using the Clean Soil process showed that tar oil content of less than 0.1 percent in clean soil can be obtained.

The process has a good potential for remediation of soils contaminated with tars, oils and variety of oil spills. Work on further refinement of the process is being continued.

3. Design Studies of Separation Mechanisms

Comparison of Batch and Continuous Unit Tests

Figure 3.1 depicts the results of site #1-1 testing in batch and continuous modes of operation using mixing vessels from the IATF. The batch tests include results from two tumblers, 6.5 liter operating volume (small) and 120 liter operating volume (large), from the IATF operated in batch configuration. The graph shows a rapid reduction of the tar content in the treated soil during the first 5 to 10 minutes of agitation, which is followed by a gradual leveling of the tar concentration at longer tumbling times. The average tar concentration for the treated soil ranged between 2 to 2.7 percent.

The IATF continuous runs are depicted in Figure 3.1 for four size fractions of the cleaned soil. The tar concentration ranged between 1 to 3.5 percent but it varied significantly between different size fractions, partly because of the presence of large particles of coke and other carbonaceous materials in the contaminated soil. There is also an apparent difference in performance between fine ($d < 1$ mm) and coarse materials. Comparison of the batch and continuous tests suggests that the test results are similar for the tumbling times of 20 and 16 minutes.

Critical Parameters in Major Process Steps

The following section lists critical parameters and process issues which require further study for optimization of the Clean Soil process. These issues were not addressed in the current program because of the scope and schedule limitation of the project. The issues are, however, recommended for future process development work.

1. Studies of the tumbling mechanisms including the effect of time, solids concentration, temperature and tar/coal ratio.
2. Studies of the required separation cuts (screens) after tumbling as a function of tar distribution in different size fractions of contaminated soils.
3. Studies of high shear mixing mechanisms and the effect of bridging oil concentrations and solid concentrations.
4. Studies of the reprocessing step including development of the approach and optimization techniques for reprocessing of different soil samples.

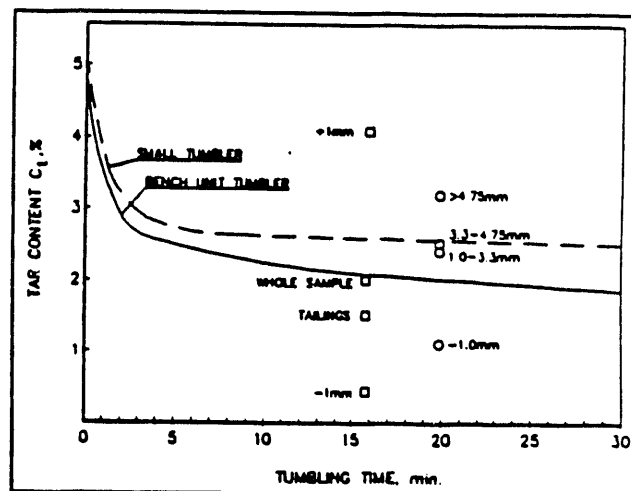


Figure 3.1. Results of contaminated soil cleanup for batch and continuous tests.

○ IATF Run No. 4, □ IATF Run No. 9.

4. Integrated Agglomeration Test Facility (IATF)

4.1. IATF Design for the Clean Soil Process

The overall objective of the IATF was to conduct process research and engineering studies on:

- beneficiation of low rank coal by de-ashing and moisture reduction (the Agflotherm process),
- de-sulphurization of bituminous coal through pyrite removal (the Agfloat process), and
- the cleaning of hydrocarbons from contaminated soils (the Clean Soil process).

For each of these processes, the IATF was used to:

- verify the developed agglomeration processes in the smallest possible continuous unit,
- identify the factors which would be used in scaling-up the process for commercialization,
- obtain engineering and operation data required for process evaluation and larger plant development, and
- develop process control philosophy.

4.1.1. IATF Design Specifications

The facilities included all major equipment needed for flow-sheet configurations studied in support of the Clean Soil process. Equipment needed for the primary and secondary crushing of coal and for the dewatering of agglomerates was not included.

The nominal IATF design specifications were as follows:

Nominal Plant Capacity: 0.25 tph or 6 t/day of coal or soil treated.

Process Configuration: Various flowsheet configurations as required in process development research.

Contaminated Soil System: 250 kg/h; feed refuse size 50 mm; coarse reject >1 mm top size.

Coal Grinding System: 250 kg/h; feed coal size <10 mm; pulverized size variable.

Agglomeration Step: High shear mixer, 58 liters capacity, with 4 minutes mean residence time; low shear mixer, 500 liters capacity, with 26 minutes mean residence time; total slurry flow – 750 to 950 kg/h.

Flotation Cells: 410 kg/h of froth.

Floor Space: Three levels, 63 sq. meters each.

Utilities: Water, 2.5 tonnes per hour electricity, 120 amp, 575 V.

Plant Operation: 8 to 10 hour test periods extended up to 72 hours.

Equipment: Waterproof, electrical enclosures meeting NEMA 12 standard or equivalent.

The following sections describe the equipment used, the work performed and the design methods developed in the IATF.

4.1.2. IATF Flowsheet

Reference flowsheet #ARC20 presents the IATF configuration for the Clean Soil process. The facilities include the following unit operations:

- contaminated soil tumbling,
- separation of coarse soil,
- coal grinding and slurry preparation,
- oil preparation,
- microagglomeration,
- flotation,
- macroagglomeration,
- screening,
- waste treatment, and
- process control.

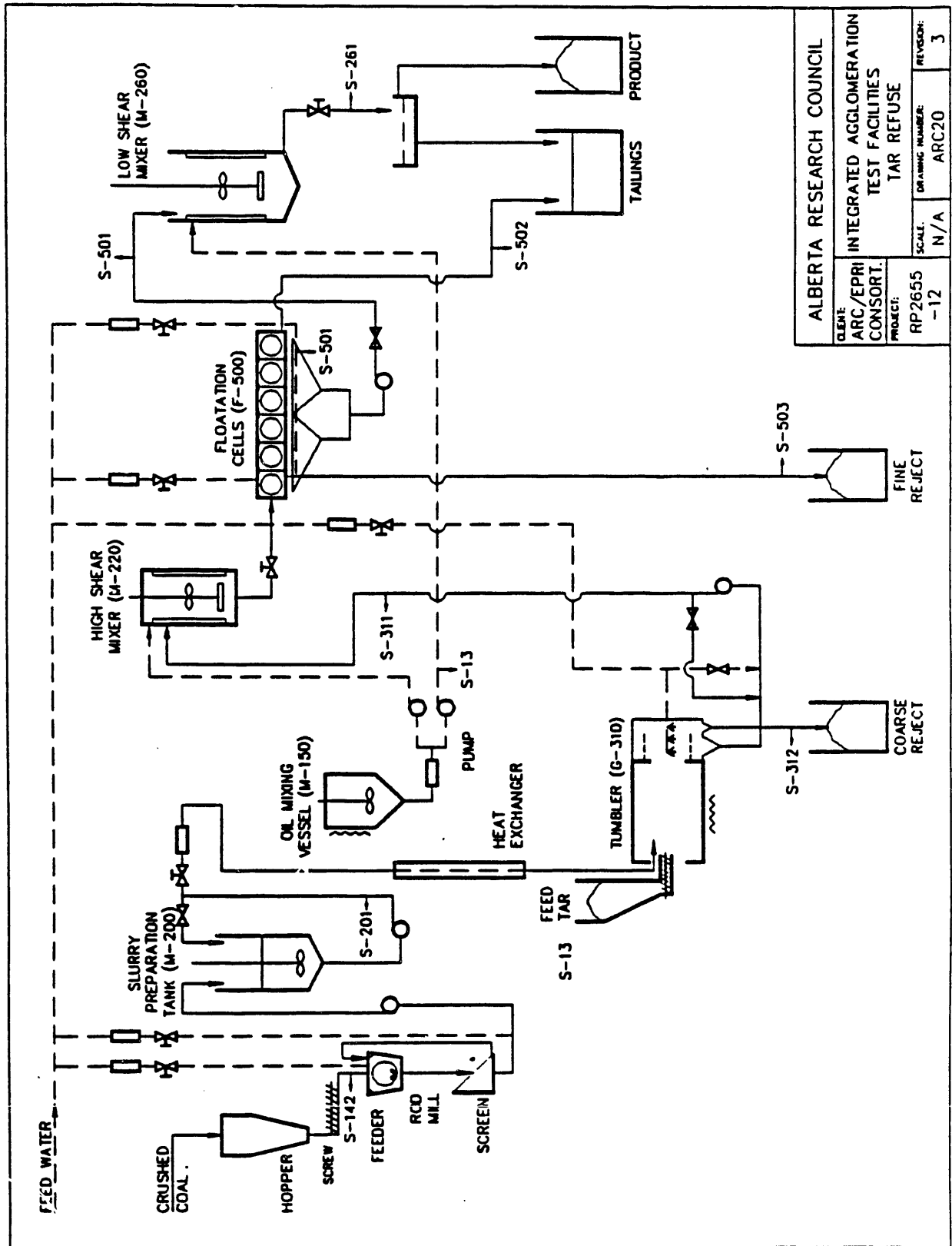
4.1.3. Major Equipment and Instrumentation

Contaminated Soil Tumbling: The tar refuse tumbling system contains a hopper and an auger, a tumbler drum, a trommel-screen attached to the drum, and a sump pump.

The hopper-screw feeder is a 35 cubic foot capacity, Thermo Design system with a maximum feed rate of 300 kg/hr. The screw feeder has a 150 mm diameter and a variable speed control. The auger's screw is extended into the tumbler and is powered by a 1.0 hp motor. The hopper is made of carbon steel.

The design of the tumbler was based on that of pilot tumbling systems used for hot water extraction of bitumen from tar sands. It is equipped with a 7.5 hp motor and a gear-train assembly. The tumbler's drum is 813 mm in diameter, 1220 mm in length with about 120 liters of operating capacity and dual opposing discharge scoops. The tumbler's rotation can be varied from 0 to 32 rpm. For heating of the tumbler's slurry charge, a 25 kW electric heater is provided.

The tumbler discharge is equipped with a trommel-screen, (12.5, 3.2, and 1.0 mm apertures) to separate coarse materials and discharge those materials to a drum. The sump pump for the screen underflow is equipped with a 5.0 hp MINPRO pump.



ALBERTA RESEARCH COUNCIL			
CLIENT:	INTEGRATED AGGLOMERATION TEST FACILITIES		
PROJECT:	TAR REFUSE		
PROJECT NUMBER:	RP2655	DRAWING NUMBER:	ARC20
SCALE:	N/A	REVISION:	3
	-12		

A detailed description of the remaining equipment of the IATF is included in Section 4, Volume 1 of this report. The following summarizes just the key equipment used in different IATF sections.

Coal Grinding and Slurry Preparation: This section contains a coal hopper and feeder system, a rod-mill, a vibrating Derrick coal screen, a coal slurry preparation tank, a drum tipper, a sump pump and two water storage tanks.

Coal slurry is preheated in a steam heated, counter current heat exchanger.

Oil Preparation and Handling: The system includes and electrically heated and agitated oil-mixing vessel, a hoist and crane for receipt of heavy oil and solvent drums, and an oil pump.

Microagglomeration: Equipment in the microagglomeration section consists of a high shear mixer, and a sump pump.

Floatation: A DENVER Sub-A floatation cell and underflow sump pump are used in the floatation section.

Macroagglomeration: This section has one low shear mixing vessel.

Separation: A double-decker rinse and drain screen, a tailings tank and a microagglomerates receiving drum, are used in the separation section.

Waste Treatment Section: Equipment in the waste treatment section consists of a settling tank, a skimmer for removal of solids, and a bag filter in the recycle water line.

Instrumentation

The IATF has the following instrumentation in support of the Clean Soil process studies:

- fifteen sampling points, as indicated on reference flowsheet #ARC20;
- temperature measurement sensors: two K-thermocouples per each mixer, one thermocouple in the oil tank, and one thermocouple in the tumbler; and
- flow measurement instruments, to monitor the flow of rod-mill water, the flow input to the high shear mixer, and water dilution to the high shear and floatation cells.

Descriptions of the sampling points, their locations, the sizes of the samples taken and the types of analyses performed are given in Table 4.1. The rationale for and objectives of the sampling and measurement instrumentation are discussed in detail in Volume 1, Section 4 of this report.

The key steps to optimizing the performance of the process were:

- Ensuring adequate measurement and control of the soluble tar to coal ratio and of the solid and oil concentration in the high shear mixer.
- Ensuring adequate measurement and control of the slurry flow, slurry solid concentration and the bridging oil plus soluble tar in the low shear mixer.
- Measurement of the floatation underflow refuse stream to determine the amount of solids.
- Adjustment of the bridging oil fed into the low shear mixer to correct for the solids in the floatation cell tailings.

Table 4.1. Summary of sampling procedures.

I.D.	Description	Frequency (approx.)	Amount	Analysis
S-140	Feed Coal	2 hr	1 L	Prox, Cal Val
S-171	Coal Slurry (before screen)	2 hr	1 L	PSD
S-200	Coal Slurry	1/2 hr	2 x 1 L	PSD, % Solids
S-13	Contaminated Soil Feed	1-Barrel	1 kg	Prox (M.A), Tar
S-312	Tumbler Coarse Reject	1/2 hr	1 L	M.A, Tar
S-311	Coal/Tar Slurry	1/2 hr	2 x 1 L	% Solids, Ash, Tar
S-501	Flot Froth	1/2 hr	2 x 1 L	% Solids, Ash, Tar
S-502	Flot Tailings	1/2 hr	2 x 1 L	% Solids, Ash, Tar
S-503-1	Flot Bottoms	15 min.	1 L	Tar, Ash
S-503-4			1 L	
S-2E1	Low Shear Disch	1/2 hr	2 x 1 L	PSD, % Solids, Ash
S-511	Dewatered Agglom.	1/2 hr	1 L	M, ASM, Cal Val
S-600	Screen Underflow	1 hr	1 L	% Solids, Ash

Total Weight for: Feed coal, soil, oil, coarse reject, flot bottoms, agglomerates.

It should be noted that the control strategy described above is difficult because the rheology of the agglomerate slurry in the mixers is quite different from the rheology of coal-water slurries. The agglomerate slurries are strongly non-Newtonian, and it is difficult to predict the slurry behavior which depends not only on the solid concentration in the mixers but also on oil concentration and the stage of development of the agglomeration process. An accurate adjustment of the bridging oil to solid ratio in the high and low shear vessels is difficult to establish.

Process Control

The IATF's process control and data acquisition hardware includes:

1. SAFE 800 PC 8253 Processor for flow and level control.
2. VAX 750 for data base management and reporting.

A brief description of the equipment is provided in Appendix 4, Volume 1 of this report.

4.1.4. Plant Safety and Environmental Issues

A hazard risk assessment was conducted for the IATF to identify potential safety risk factors in the operation of the plant. In general, the plant operation was classified as relatively low risk since it involves low temperatures and pressures and hydrocarbons with low volatility. Exceptions to this general classification were:

- generation of coal dust in the coal grinding area, and
- potential equipment hazards such as sparking in the fan handling dust, high noise levels, staff being caught up in drives or burnt by hot equipment, etc.

Risk from these potential hazards was reduced by installation of a non-sparking fan for handling dust, installation of guards for all rotating equipment components, and insulation of hot pipes and equipment. Special enclosures were installed around the rod-mill to reduce the noise levels. The protective measures taken, the equipment installed and the environmental review conducted are described in detail in Section 4 of Volume 1.

4.2. Plant Operating Experience and Optimization

4.2.1. Plant Commissioning and Results

Plant commissioning for the Clean Soil process started on September 1, 1989, after IATF equipment was tested for the Agglomer process configuration. A number of equipment tests were performed in support of the IATF. These are summarized in Table 4.2. Tests conducted specifically for the tar refuse samples were:

- **Tumbler Circuit Improvement Tests:** An automedium cyclone was installed to reduce the amount of coarse sand in the feed to the high shear mixer. The cyclone was later disconnected.
- **Initial Process Verification Tests:** A supplementary bridging oil was delivered to the high shear mixer in order to improve the efficiency of the floatation cell. Floatation with refuse only resulted in small agglomerates and low floatation efficiency.

The commissioning of the IATF for refuse tar clean-up was completed in October, 1988 when several continuous runs were conducted.

Following the commissioning, the tests were conducted with bituminous coal to prepare 5 to 6 tonnes of agglomerates for combustion tests. The tar refuse sample was a Site #2 blend with about 7 percent tar. In total, 50 drums of agglomerates with 5.7 percent ash were produced over 120 hours of plant operation. The longest uninterrupted period of operation extended for 28 hours. The operation-related observations during the unoptimized runs were:

- Because the plant was new and not fully debugged, it was difficult to maintain steady-state plant conditions.
- Failure of equipment resulted in the overflow of agglomeration vessels and in line plugs.
- Pilot-plant waste treatment and disposal was a problem.
- Plant capacity was less than 50 percent of the installed plant capacity of 6 tonnes per day due to the above mentioned problems.

4.2.2. Equipment Studies and Modifications

Major equipment modifications related to plant operation in support of the Clean Soil process and other processes are presented in Volume 1, Chapter 4 of this report. The equipment modifications conducted specifically for processing tar refuse are described in the sections which follow:

Tumbler System

The following were the modifications introduced to the tumbler system:

- A new 24 kW electric heater was installed under the tumbler to improve the heating capacity of the system.
- Two thermocouples were installed, one in the inlet and the second in the outlet of the tumbler to monitor the temperature of the feed and discharge slurry.
- The tumbling frequency was changed to 15 rpm to improve the mixing and handling of the materials.
- The tumbler's motor power was increased from 5 hp to 7.5 hp.

4.2.3. Instrumentation and Control System

Most of the instrumentation performed as expected from the design specifications. However, difficulties were encountered in the development of a workable process control strategy because of the system's instability. The main process control problems were:

- lack of feedback information on variation in moisture and ash content of the feed coal;
- unreliable measurement and fluctuations in of the coal slurry flow rate and slurry solid concentration, which often led to formation of soft agglomerates and plugging of the mixers and mixer outlets. Also lack of accurate measurement of slurry flow and solid concentration often resulted in oil over-dosage; and
- fluctuation of product discharge for the floatation cells.

The first six months of operation of the IATF at non-optimized conditions, resulted in production of large samples of products and in a better understanding of the performance of the process, includ-

Table 4.2. Summary of plant commissioning tests.

Tests	Purpose	Observations	Modifications
Water Tests	To test all pumps, valves and lines	Various leaks at joints, plugs in lines	Leaks corrected, lines flushed
Sand-Water Tests	To check coal hopper-feeder and sand pump performance, line capacities and solids handling	Performance poor, coal feeder marginal with wet sand	Sand-pump drives modified, vibrator installed on feeder
Coal-Water Tests	To test rod-mill performance, pump and line capacities, to check process control and instrumentation	Over-grinding of coal in the rod-mill, poor performance of Moyno pump. Controls instruments OK.	Rod charge adjusted, Moyno replaced with sand pumps
Coal Grinding Tests	To optimize grinding conditions	See Section 3.1, Volume 1	Optimized rod charge
Agglomeration Tests	To test Agflotherm circuits, mixer performance, floatation cell and screen capacities, to check the oil feed system	Various other plant configurations achievable by of lines, low shear mixer performance poor, floatation cell froth discharge inadequate, screen blinding serious	Slight changes, additions to the slurry lines, low shear mixer impellers modified, froth removal paddles extended, welded wire screens ordered

ing process control required. Evaluation of existing problems and identification of new solutions and design concepts were significant benefits of operating the plant round-the-clock. Process control improvements identified during the six months of operation will be implemented in the next phase of the process development work.

4.2.4. Pilot Plant Optimization

Following commissioning and equipment modification, the IATF was operated to test the equipment configuration for the clean-up of tar refuse samples. Selected IATF runs for cleaning tar refuse from site #1-1 are described below.

Run No. 6

The test was conducted at 50 percent plant capacity to determine the following process and operational parameters:

- mass balance around the major equipment,
- tar concentration in various process streams,
- performance of the tumbler heating and temperature control system, and
- operation of the floatation cell soil removal system.

The test was completed as scheduled without a shutdown. Detailed data for the run are enclosed in Appendix 3. The key conclusions from the test results were:

- About 41 percent of the contaminated soil appeared as a coarse reject ($d > 1.0$ mm), 45.3 percent appeared as fine reject from the floatation cell, and 13.7 percent as combined tailings (Figure 4.1).
- The soluble tar concentration varied considerably among process streams. The coarse reject had about half of the soluble tar concentration (2.8 to 5.0 percent vs 5.8 percent in feed), the fine reject had soluble tar concentration in the range of 0.99 to 1.41 percent (Figure 4.2).
- The average soluble tar concentration in tailings from the floatation cell was 1.5 percent (Appendix 3).

In summary, the run was considered successful and preparations were made for the IATF operation at 100 percent capacity.

Run No. 7

The primary objective of the run was to determine the tar content in each stream of the IATF at 100 per-

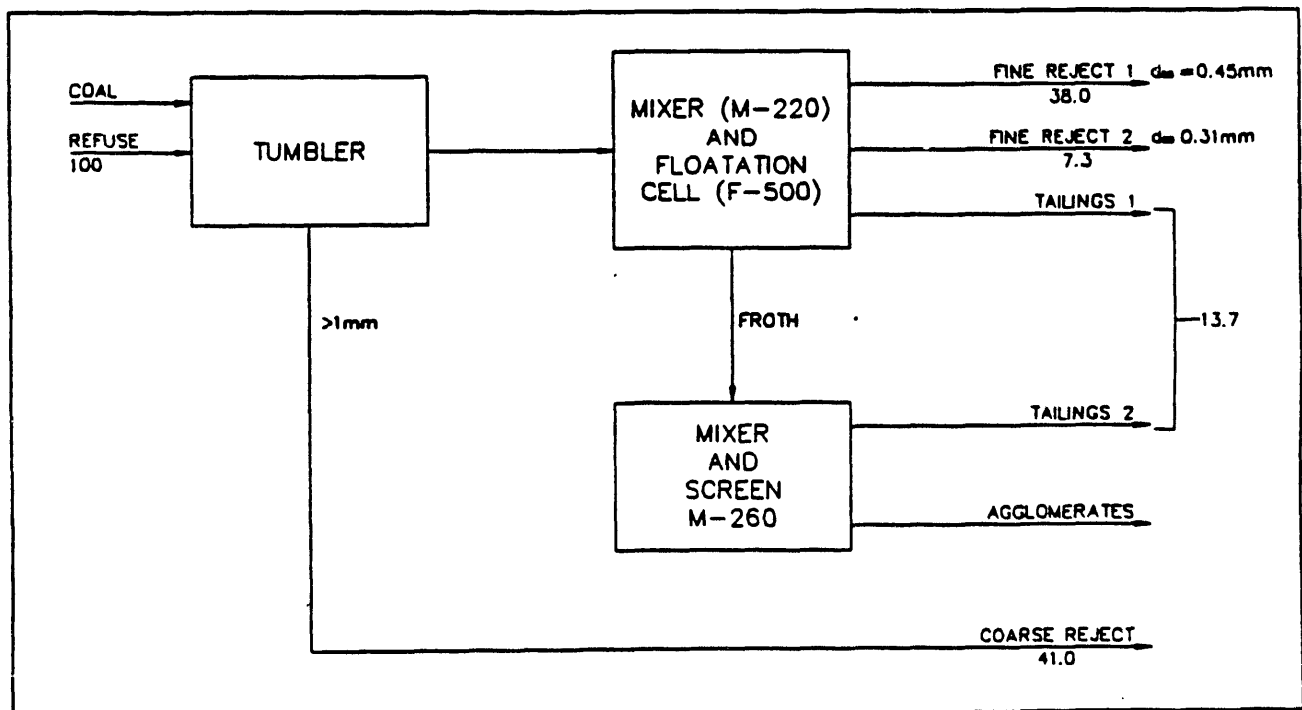


Figure 4.1. Overall material balance for the cleaned soil (Run 6).

cent plant capacity. The planned tests proceeded satisfactorily despite several minor equipment malfunctions, higher than planned coal feeding rate, overheating of the low shear vessel drive motor, etc. The run was completed without any interruption and the test results suggested that there were significant differences in the concentration of soluble tar in the process streams (Figure 4.3 and Table 4.3). This would suggest that more detailed information about tar levels in different streams was needed and that the amount of coal should be reduced.

Run No. 8

Two changes were introduced in Run 8, i.e., the system was operated at a higher tar/coal ratio (12 to 14 vs 7 to 8 in Run 7) and a sampling system was incorporated in the floatation cell to provide information on variations in fine refuse composition.

Also, because startups and shutdowns result in the removal of a significant amount of coal with soil fines in floatation tailings, improved IATF operating procedures minimizing these effects were introduced. As a result of the operational changes introduced, the fine soil removed from the floatation cell was cleaner than the soil removed in Run 7 (0.68 and 0.38 percent of tar oil vs 0.76 and 0.66 percent

Table 4.3. Concentration of soluble tar in the feed and products (percent, dry basis).

Run No.	Feed	Coarse reject		Fine reject	
		$d > 1.0\text{mm}$	$d_{50} = 0.45$	$d_{50} = 0.31$	Tailings
7	5.6	3.4 - 4.1	0.76	0.60	1.8
8	6.3	4.3	0.68	0.38	-
9	5.8	3.5 - 4.7*	0.52	0.34	1.6

* corrected for mass balance.

of tar oil for Run No. 7) (Figure 4.4 and Table 4.3). The results also suggested that further improvements may be possible in the efficiency of cleaning of the tar refuse.

Run No. 9

The objectives of Run 9 were to operate the IATF with improved feeding of coal slurry (runs 6 to 8 experienced some difficulties with coal slurry feeding), and to establish performance limitations for the process. The results obtained are depicted in Figures 4.5 to 4.8 and Table 4.4. The key features depicted by the figures and tables are:

- The soluble tar concentration in the fine reject and tailings was relatively constant and unaffected by

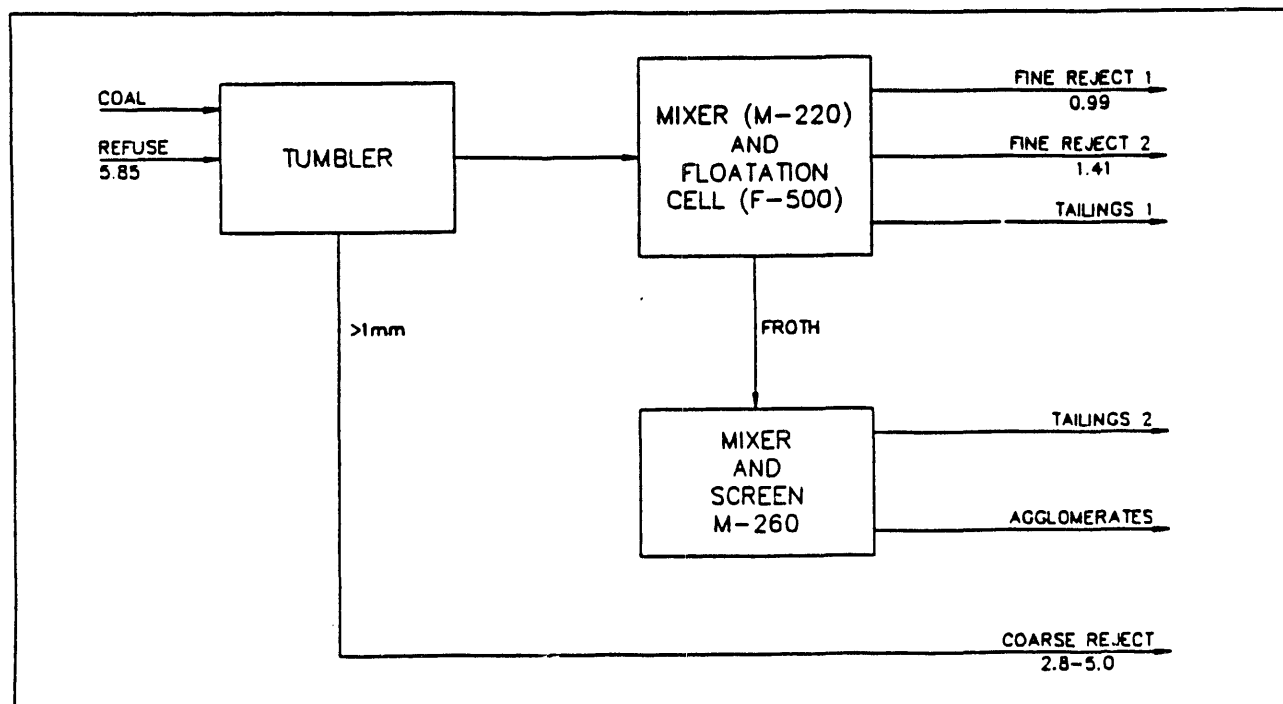


Figure 4.2. Concentration of soluble tar in feed and products (Run 6).

variations in soluble tar concentration in the feed (Figure 4.5).

- The concentration of soluble tar in the fine reject from flotation cell #1 was high at the run startup, but then it quickly stabilized at about 0.5 percent (w/w dry basis, Figure 4.5).
- The change in the feed soluble tar concentration, or its size consistency, affected the soluble tar content in the coarse reject (Figure 4.5).

Table 4.4. Distribution of soluble tar in the products (Tar Oil in Feed=100).

Run No.	Coarse reject		Fine reject		Tailing 1 & 2	Coal agglom.
	d>12mm	d50=0.45mm	d50=0.31mm	d50=0.25mm		
8	36.3	2.4	0.4	2.0	59.0	
9	30.6	2.5	0.4	1.9	64.6	

- The majority of the refuse (soil and soluble tar) was distributed between the coarse reject (36.00 percent), fine reject ($d_{50}=0.45$ mm, 21.0 percent) and agglomerates (29.7 percent) (Figure 4.6, Table 4.4).
- The soluble tar was distributed mainly between the agglomerates (64.6 percent) and the coarse reject (30.6 percent). Fine refuse and tailings contained only about 4.7 percent (combined) of soluble tar (Figure 4.7, Table 4.4).
- The concentration of soluble tar (w/w dry basis) was 13.5 percent in the agglomerates, 5.2 percent in the coarse refuse, 1.6 percent in the combined tailings, and less than 1 percent in the fine refuse. This compares to 6.2 percent of soluble tar in the feed refuse (Figure 4.8).

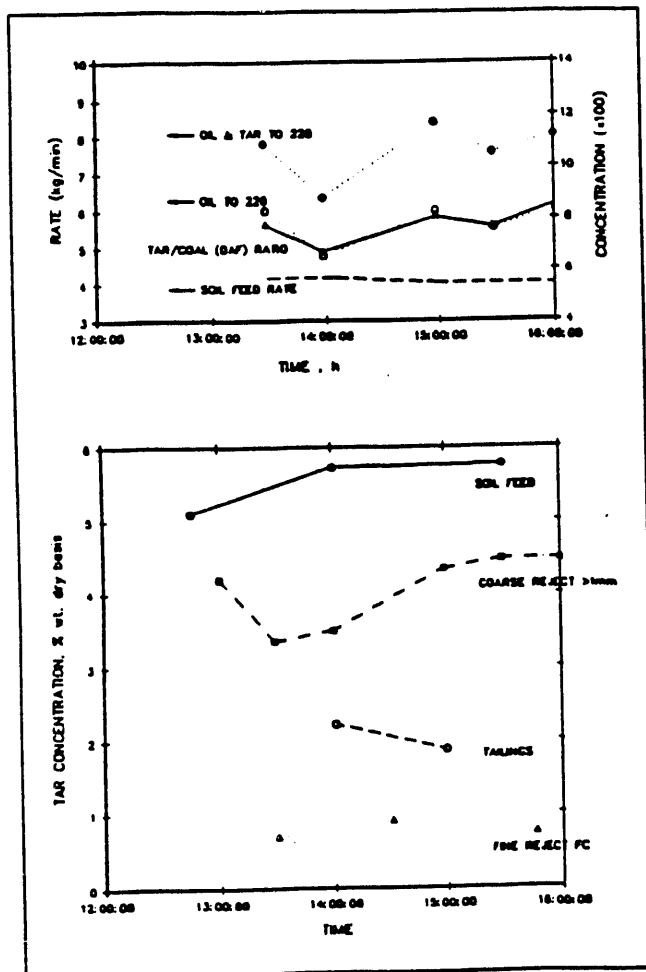


Figure 4.3. IATF Run No. 7 (process variables and soil streams).

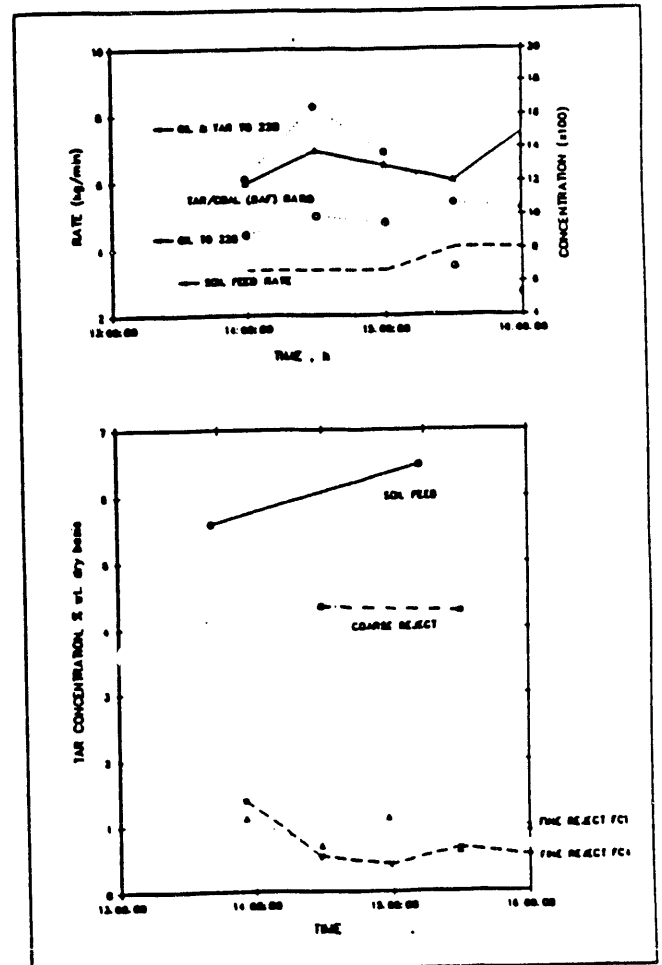


Figure 4.4. IATF Run No. 8 (process variables and soil streams).

Summary of Runs 7 to 9

Tables 4.3 to 4.5 provide a summary of mass balance, of soluble tar concentration and tar distribution for runs 7,8 and 9. These tables show that as the runs were progressively improved for feeding refuse from site #1-1:

- The concentration of soluble tar in the coarse and fine rejects, and tailings remained unaffected (Table 4.4).
- The amount of soluble tar capture increased in the coal agglomerates and decreased in the coarse reject fraction. Tar content in the fine reject remained unchanged, however (Table 4.5).
- Despite the decrease of tar content in the coarse reject, the amount of the coarse reject remained unchanged (Table 4.6). The increase of the soil content in the tailings and agglomerates was at the expense of the fine reject ($d_{50}=0.4$ mm) fraction.

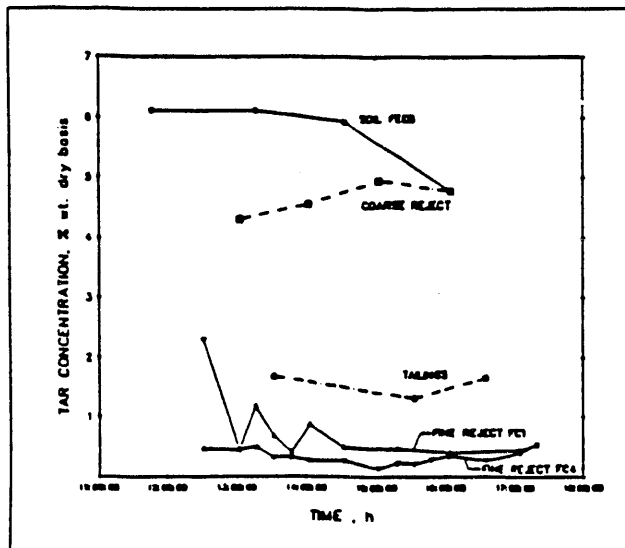


Figure 4.5. IATF Run No. 9 (soil streams).

Table 4.5. Overall mass balance for tar refuse (Feed=100).

Run No.	Coarse reject $d > 1.0\text{mm}$	Fine Reject $d_{50} = 0.4\text{mm}$	Fine Reject $d_{50} = 0.22\text{mm}$	Tailings and agglomerates
7	34.0	30.0	7.1	28.9
8	37.0	22.3	6.4	34.3
9	36.0	21.0	5.9	37.1

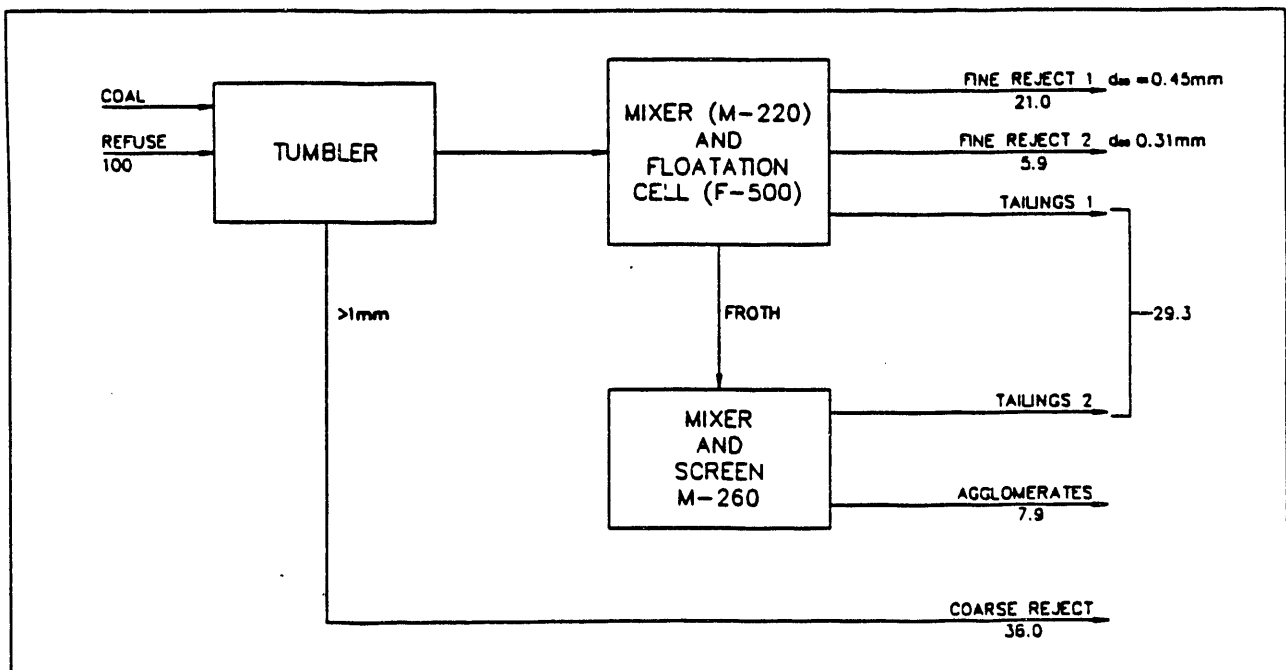


Figure 4.6. Overall material balance for tar refuse (Run 9).

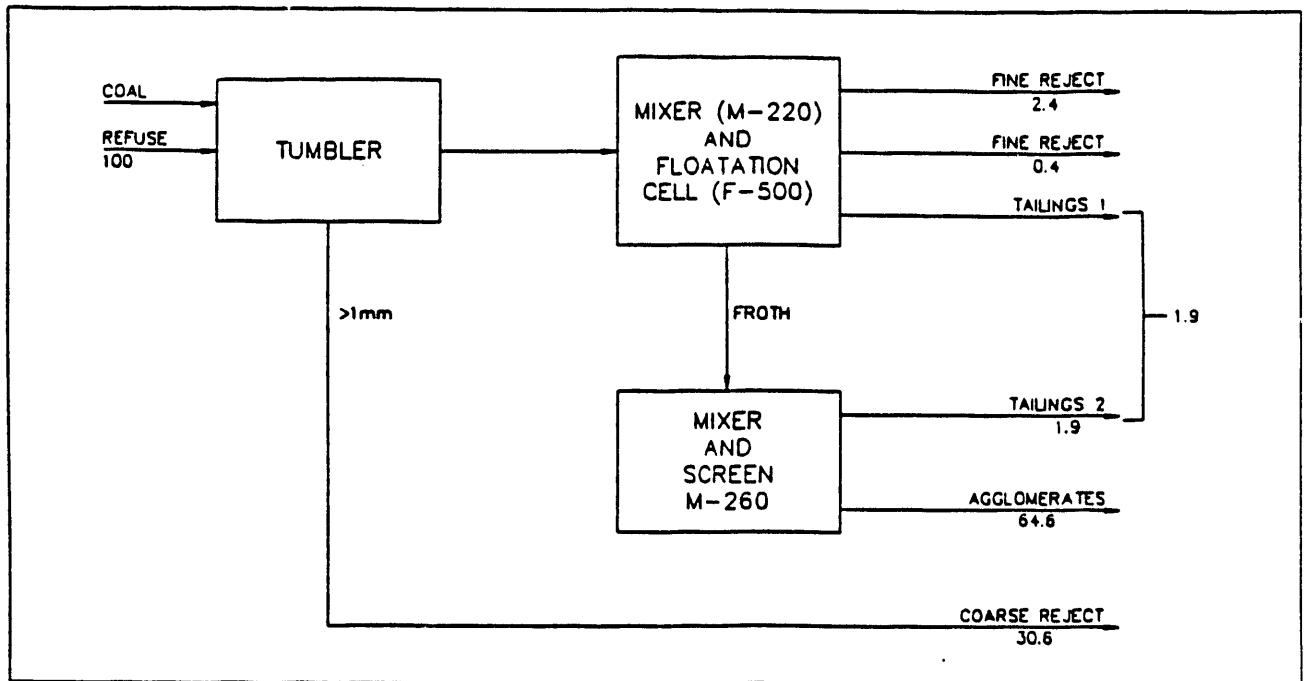


Figure 4.7. Distribution of soluble tar in different products (Run 9).

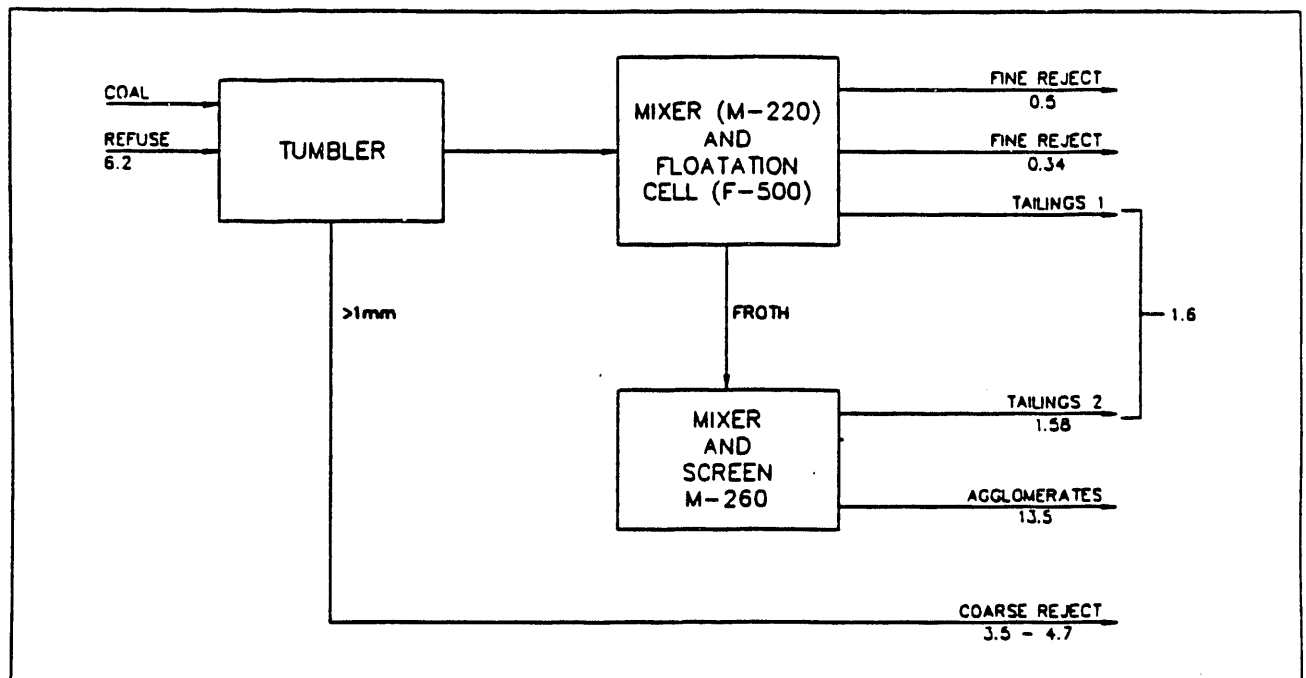


Figure 4.8. Concentration of soluble tar in feed and products (Run 9).

4.3. Conclusions

The specific objective of the Integrated Agglomeration Test Facility was to obtain engineering and operation data required for the Clean Soil process evaluation and development, to identify the factors which are important in the scaling-up of the process, and to develop process control and optimization methodology. The facilities included all equipment in the configuration representing the Clean Soil process, with the exception of the primary and secondary coal crushers.

The major process steps and equipment included contaminated soil tumbling and coarse soil separation, coal grinding of slurry preparation, bridging oil preparation, microagglomeration and floatation, macroagglomeration and screening of large agglomerates, and waste treatment. In addition to the temperature and flow measurement instrumentation, the IATF had fifteen sampling points for slurry and product streams.

The keys to improving the process performance were the measurement and control of the soluble tar to coal ratio, the solid concentration in the high shear mixer, the amount of the bridging oil plus soluble tar in the low shear mixer, and the amount of

solids in the floatation underflow. Deviation of these parameters from their preferred values often resulted in a rapid decline in process performance. In order to improve process controllability and performance, a number of equipment modifications were introduced in the course of the program. The most important were installation of an electric heater and temperature control on the tumbler, and improvement of the control instrumentation. As a result of these changes and process optimization the tar content in fine soil reject ranged from 0.95 - 1.41 percent to 0.34 to 0.52 percent in floatation cells #1 and #4, respectively.

The process improvement in Runs No. 6 to No. 9 increased the performance of the process. Specifically, the soluble tar content increased in the coal agglomerates and decreased in the coarse reject fraction. However, the concentration of soluble tar in reject was still relatively high (0.35 to 0.5 percent) despite the fact that five refuse and tailings contained only about 5 percent of total soluble tar. Therefore, further revisions have been recommended which should produce results as favorable as those previously demonstrated in bench scale equipment.

5. Product Testing and Characterization

5.1. Agglomerates Testing and Characterization

Approximately 3.5 tonnes of agglomerates were produced in the IATF using tar refuse from Site #2 and high volatile Eastern U.S. bituminous coal (HVB). The agglomerates (MGP2) were tested at Combustion Engineering, Inc., Hartford, Connecticut, for their handling and combustion performance in 25 to 100 percent blends with the HVB Coal.

A summary of the contractor's major findings and excerpts from the reports appear in the sections which follow.

Table 5.1. Analysis of MGP2 and HVB coal.

	HVB COAL		MGP2	
	As received	Moisture free	As received	Moisture Free
Proximate (wt, %)				
Moisture (Total)	4.6	-	22.8	-
Volatile Matter	32.7	34.3	29.5	38.2
Fixed Carbon (diff)	54.2	58.8	43.2	58.0
Ash	8.5	8.9	4.5	5.8
Total	100.0	100.0	100.0	100.0
Ultimate (wt, %)				
Moisture (Total)	4.6	-	18.4	-
Hydrogen	5.0	5.1	4.5	5.5
Carbon	74.5	78.1	65.9	80.8
Sulphur	0.8	0.9	1.0	1.2
Nitrogen	1.5	1.6	1.2	1.5
Oxygen (diff)	5.1	5.4	3.4	4.2
Ash	8.5	8.9	5.6	6.9
Total	100.0	100.0	100.0	100.0
HHV (Btu/lb)	13,256	13,895	11,791	14,750
HHV in Volatile				
Matter (Btu/lb)	5,750	6,027	5,607	6,872
lb ash/MBtu	6.4	-	4.7	-
Flammability Index				
(°F)	a	-	a	-
Ash Fusibility (RED.ATM)^a				
IDT	-	2,010	-	2,000
ST	-	2,700+	-	2,670
HT	-	2,700+	-	2,700+
FT	-	2,700+	-	2,700+
(FT-IT)	-	690	-	700+
Ash Composition (wt, %)				
SiO ₂	-	57.9	-	53.2
Al ₂ O ₃	-	29.9	-	30.4
Fe ₂ O ₃	-	5.2	-	7.7
CaO	-	2.1	-	2.3
MgO	-	0.6	-	1.0
Na ₂ O	-	0.3	-	0.4
K ₂ O	-	1.2	-	1.5
TiO ₂	-	2.0	-	1.8
So ₃	-	0.8	-	1.4
Total	-	100.0	-	99.8

a: Analysis in progress.

5.1.1. Handling and Storage

An analysis of the HVB coal and MGP-2 sample is shown in Table 5.1. Both fuels have similar proximate and ultimate analyses and their ash fusion characteristic temperatures are very close.

Table 5.2 presents results of Hardgrove Grindability tests for HVB Coal, the MGP2 sample and their blends. Results show compaction for blends with 50 percent or higher content of MGP2, and a higher Hardgrove Grindability index for the 25 percent blend, as compared to the HVB coal sample.

In general, analyses of the agglomerates and the sample coals indicate that the fuels have the following characteristics:

- Both fuels have ashes with high fusion temperature and are likely to display similar ash slagging and fouling behavior.
- Blends of MGP2 and HVB coal samples exhibit slight to moderate compaction at 50 percent and higher concentration of MGP2.
- Moisture in the MGP2 sample appears to enhance the compaction of the agglomerate samples.

5.1.2. Agglomerates Combustion

Figure 5.1 depicts a schematic representation of the Combustion Engineering pilot-scale combustion rig in Hartford, Connecticut. The specific product characteristics evaluated in the pilot-plant combustion tests were:

- general grinding characteristics
- fuel feeding
- fireside performance
- furnace slagging characteristics
- convective pass fouling characteristics
- fly ash erosion
- gaseous and particulate emissions.

Tables 5.3 to 5.8 present the results of the pilot scale performance evaluation for a 25 percent

Table 5.2. MGP2 Hardgrove grindability test results.

Sample	Moisture content (%)	Grinding characteristics	HGI
100% Coal	4.6	Good	42.5
25% MGP2*	7.1	Good	48.5
50% MGP2	7.1	Slight Compaction	-
75% MGP2	7.1	Slight Compaction	-
100% MGP2	7.1	Moderate Compaction	-
100% MGP2	22.8	High Compaction	-

* Balance HVB coal.

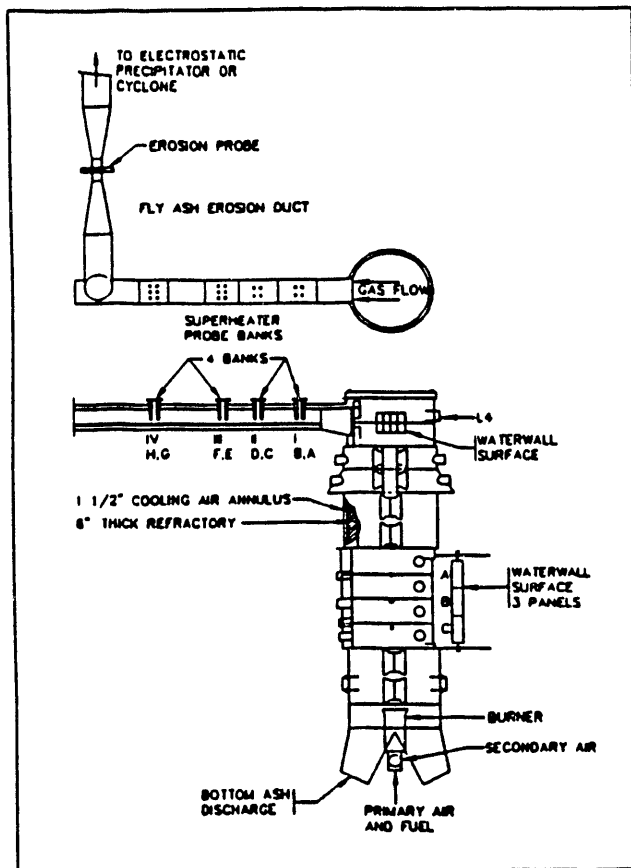


Figure 5.1. Fireside performance test facility at Combustion Engineering, Inc.

Table 5.3. Preliminary pilot scale bowl mill pulverization results.

Test parameters	0% MGP2*	25% MGP2*	50% MGP2*
Mill Outlet Temperature, °F	150	150	150
Mill Power Consumption, kWh/MBtu	0.67	0.63	0.64
Mill Classifier Settings	1 1/3	1 1/3	1 1/3
Fuel Fineness, wt %	71.2	69.2	67.3
Compaction/Pasting	None	None	None

* balance HVB coal.

Table 5.4. Pilot scale combustion test conditions.

Firing conditions	25% MGP2 Blend*			100% Coal
	Test 1	Test 2	Test 3	Test 4
Firing Rate (MBtu/hr)	4.0	4.1	4.2	4.2
Excess Air (%)	20	20	20	20
Furnace Temp (°F)	2960	2990	3050	3040
Superheater Temp (°F)	2410-1910	2410-1910	2430-1980	2450-1970

* balance HVB coal.

Table 5.5. MGP2 convection fouling characteristics at 4.2 mbtu/hr firing rate.

Parameters	25% MGP2*	100% Coal
Deposit to tube bonding strength	3	3
Soot blowing frequency, hr	8 to 10	8 to 10
Deposit physical state	Lightly Sintered	Lightly Sintered

* balance HVB coal.

Table 5.6. Chemical composition of ash deposits generated at 4.2 mbtu/hr firing rate.

	100% Coal			Fly Ash	25% MGP2 Blend			Fly ASH
	As-Fired Fuel Ash	W.W. Ash	S.H. Ash		As-Fired Fuel Ash	W.W. Ash	S.H. Ash	
Ash Fusibility, °F								
IT	2150	2260	2240	2180	2160	2160	2240	2230
ST	2700*	2440	2570	2360	2700*	2700*	2650	2570
HT	2700*	2670	2620	2640	2700*	2550	2700*	2700*
FT	2700*	2700*	2700*	2700*	2700*	2700*	2700*	2700*
FT - IT	550	440	460	520	540	540	460	470
Ash Composition, wt %								
SiO ₂	58.4	59.8	58.6	58.6	61.4	56.3	60.9	59.0
Al ₂ O ₃	26.7	28.5	29.3	26.7	25.2	28.4	29.0	28.2
Fe ₂ O ₃	7.6	7.3	6.1	8.0	7.1	9.0	5.9	6.9
CaO	1.8	1.3	1.6	2.1	1.4	2.6	1.6	1.9
MgO	0.7	0.5	0.6	0.7	0.6	1.0	0.6	0.7
Na ₂ O	0.1	0.1	0.1	0.1	0.1	0.1	<0.1	0.1
K ₂ O	1.0	0.8	1.0	0.8	0.8	1.0	0.1	1.0
TiO ₂	1.7	1.6	1.6	1.6	1.4	1.6	1.8	1.7
So ₃	2.0	0.1	0.1	1.3	2.0	0.1	0.1	0.5
Total	100.0	100.0	99.0	100.0	100.0	100.0	100.0	100.0
Carbon, wt %	-	-	-	3.4	-	-	-	4.0
Mass Mean Diameter	-	-	-	10.5	-	-	-	13.4

Table 5.7. EP toxicity test results for Site 2 coal and 25% MGP2 blend.

	100% Coal		25% MGP2 blend		EPA maximum levels (ppm)
	Bottom ash (ppm)	fly ash (ppm)	Bottom ash (ppm)	Fly ash (ppm)	
Arsenic	0.7	0.7	0.7	2.2	5.0
Barium	<0.1	<0.1	<0.1	<0.1	100.0
Cadmium	<0.01	<0.01	<0.01	<0.01	1.0
Chromium	0.3	0.05	0.4	0.07	5.0
Lead	<0.1	<0.1	<0.01	<0.1	5.0
Mercury	<0.0002	0.0002	<0.0002	<0.0002	1.0
Selenium	<0.08	0.5	<0.08	0.7	1.0
Silver	0.03	<0.01	0.03	<0.01	5.0

Table 5.8. Gaseous emissions from coal and 25% MGP2 blend combustion tests.

Gaseous Emissions	25% MGP2 Blend	100% Coal
SO ₂ , ppm 3% O ₂ dry (theoretical)	757 (763)	761 (765)
NO _x , ppm	1380	1385
SO ₃ , ppm	-3	-4
Volatile Organic Matter lb/ton*	0.009	0.011
lb/MBtu fuel	3.2 x 10 ⁻⁴	4.1 x 10 ⁻⁴
Polynuclear aromatic hydrocarbons 10 ⁻⁶ lb/MBtu fuel	ND	ND

* EPA published emission rate for coal combustion sources = 0.07 lb VOC/ton. (Compilation of air pollutant emission factors - EPA AP-42)

MGP2 blend. In total, three firing rates were tested for the blend and one firing rate was tested for the HVB coal sample to provide a reference baseline. The key features and conclusions which can be drawn from these tests were:

- Fuel blends with up to 50 percent of MGP2 had good flowing characteristics in the pilot plant equipment (from the chute to bucket elevator, to the raw fuels storage silo, to a gravimetric feeder and to a pilot scale bowl mill).
- Grinding of the fuel blends requires slightly less mill energy than is needed for the coal sample (Table 5.3).
- Good, stable flames were obtained during a combustion run with the 25 percent blend. The

carbon content in the fly ash was low and combustion efficiencies were 99.7 percent.

- Both the 25 percent blend and the HVB coal had low slagging propensity and similar deposit accumulation and waterwall heat absorption rates (Table 5.5). Removable waterwall deposits were obtained with both fuels at full thermal load (4.2 mm Btu/hr) and at maximum flare temperature of 3050°F or more.
- Chemical composition of the coal and blend were essentially very similar (Table 5.6) and EP toxicity tests conducted on bottom and fly ashes for both fuels indicated that key trace elements were below the levels allowed by the EPA (Table 5.7).
- Analyses of gaseous emissions from combustion of both fuels (Table 5.8) showed sulphur levels close to the theoretical SO₂ emissions calculated from fuel analyses. The NO_x emissions were also similar for the two fuels.

Because the agglomerate MGP2 sample was obtained using soluble tar from tar refuse, detailed analyses were conducted for the emissions of polynuclear aromatic hydrocarbons (PAH) and volatile organic compounds (VOC). The results showed PAH levels below analytical detection limits and VOC levels below the EPA published emissions of VOC from coal combustion sources (Table 5.8). A detailed list of the PAH and VOC analyzed and their concentration levels are presented in Tables A4.1 and A4.2 in Appendix 4.

5.2. Conclusions

Approximately 3.5 tonnes of product agglomerates produced from tar refuse and HVB coal were test burned at Combustion Engineering, Inc. Hartford. The tests were conducted using 25 to 100 percent blends of agglomerates and HVB coal.

The results of handling tests showed good grinding characteristics for blends up to 25 percent of agglomerates and only slight compaction on grinding blends up to 75 percent. The mill power consumption was slightly lower for the blends as compared to the parent HVB coal.

The pilot scale combustion tests showed combustion efficiencies over 99.7 percent for 25 percent blends of MGP2 agglomerates and 75 percent of HVB coal, and good, stable flames. Both blends and HVB parent coal had low slagging propensity, similar

deposit accumulation and water wall heat absorption rates. Toxicity tests conducted on bottom and fly ashes indicated that concentrations of trace element was within a range allowable for ash disposal. Gaseous SO_2 emissions were close to the theoretical emissions predicted from fuel analyses and the

NO_x emissions were similar for blends and HVB coal. In general, blends of agglomerates and HVB coal were satisfactorily handled and combusted using conventional combustion equipment and procedures.

6. Environmental Analysis

6.1. Regulatory Issues

Section 7, "Conceptual Design and Cost Analysis of the Mobile Clean Soil Process Unit" requires that the mobile plant operation meet all 1989 emission standards in Alberta. Examples of the relevant government statutory acts and effluent limitations applicable to the mobile unit's operation are listed in Table 6.1. The key environmental control concerns with respect to plant emissions and operation are:

- Air emissions of hydrocarbon vapors from the mobile unit. The unit design calls for a "vapor eliminator" system consisting of an absorber tower with activated carbon. An alternate system consisting of a vapor incinerator can also be used where the amount of volatile hydrocarbons is small.

Table 6.1. Examples of government statutory requirements for the clean soil process.

1. Alberta Clean Air Act
2. Boiler and Pressure Vessel Act
3. Coal Conservation Act
4. Electrical Inspection and Licensing Act
5. Environmental Assessment Act
6. Fire Protection Act
7. Gas Inspection and Licensing Act
8. Ground Water Conservation Act
9. Litter Control Act
10. Mechanic's Lien Act
11. Occupational Health and Safety Act
12. Public Health Act
13. Water Resources Management Act
14. Water Rights Act
15. Alberta Clean Water Act
16. Canadian Environmental Protection Act
17. Alberta Hazardous Waste Regulations

- Aqueous discharge from the mobile unit. There is no net discharge of waste water from the unit. All waste water is recycled and reused in the process which requires a makeup water at the rate of 15 to 25 t/d.
- Discharge of leachate contaminated with hydrocarbons from the agglomerates storage pile. Agglomerates would be stored in bins from which they would be loaded on trucks. No leachate would enter the soil.
- Air emissions of hydrocarbon vapors during combustion of agglomerates. Tests by Combustion Engineering, Inc. showed that gaseous emissions from the combustion of agglomerates are similar to emissions from the combustion of HVB coal used in agglomeration. The tests showed PAH levels below analytical detection limits and VOC levels below EPA published emission limits for VOC from coal combustion.
- Disposal of ash from combustion of agglomerates. Tests by Combustion Engineering Inc. to assess the EP toxicity of bottom and fly ashes showed that key trace elements were within the limits allowed by the EPA for coal ash disposal.
- Disposal of clean soil. The level of oil remaining in the soil is projected to be below 0.1 percent. This means that the treated soil would be acceptable for land fills.

It was concluded that when decontaminating oily waste, the operation of the mobile unit would have to conform to the regulations and statutes applicable to solid waste disposal and treatment such as those applying to mine waste, etc. The situation would be,

however, quite different for decommissioning of a contaminated industrial site or for spill cleaning. In the latter case, a much broader and stricter set of requirements before, during and after operation of the mobile unit would likely have to be implemented, namely:

- A detailed review of relevant acts and regulatory statutes would be required since most provinces have no clear legislative guidelines for clean-up of contaminated sites.
- A detailed assessment of the contaminated site, the past activities on the site and its future use, type and level of contaminants, their risk and potential pathways, the site hydrology, climate, water (surface and underground) sources, etc. would be required.
- Detailed criteria for clean-up requirements, post cleaning verification, etc., including regulation approval would have to be developed for the site.

Examples of decommissioning activities required would include:

- extensive monitoring including the analyses of soil and ground water,
- excavation and reconstitution of the site,
- on-site inspection and verification of cleanup by different regulatory bodies, and
- an extensive public information program before, during and after the cleanup.

It is important to note that in all probability a combination of processes – the Clean Soil process, incineration, etc. – would be required for the most effective and efficient approach to cleaning different contaminated sites. In such cases, the amount of coal that has to be brought to the contaminated site would be much less than if only the Clean Soil process were used. For example, the part of contaminated soil which contains high tar concentrations (above 50 percent) could be more efficiently cleaned by an incineration process, while the remaining part of the soil could be cleaned using the Clean Soil process.

In summary, the requirements for the environmental analysis would vary quite significantly depending on whether the mobile Clean Soil unit was operated for cleaning oily waste from recovery and upgrading of bitumen or whether the mobile unit is used for con-

6.2. Clean-up of Oily Wastes from Heavy Oil Operations

The Problem

The production of heavy oil and bitumen in Western Canada generate large quantities of oily wastes each year. The three main areas in which heavy oil and bitumen are produced are the Cold Lake Oil Sands Area, the Lindbergh Heavy Oil & Oil Sands Area and Lloydminster Heavy Oil Area. Combined, they produced about 300,000 barrels per day of heavy oil and bitumen in 1985.

Table 6.2 shows an estimate of the quantities of oily wastes produced in these three areas in 1985. The Lloydminster Area is by far the largest waste producer, with an annual production of about 87,000 m³. Assuming a specific gravity of 1.3 for the waste, this is equivalent to about 113,000 tonnes per year. The general rule of thumb is that 0.3 to 1.0 percent by volume of wastes is generated per unit volume of heavy oil produced. As Canada will be relying more and more on its heavy oil and bitumen resources in the long run, the quantities of oily wastes produced are expected to increase rapidly.

Although most wastes from oil production operations have moderate or low toxicity, their volume is such that wise management is required to ensure safety and protection of the environment. It is recognized that current management practices, such as road disposal spreading and water flushing are an inadequate and alternative method of treatment and other methods of disposal must be sought. The Clean Soil technology which has been successfully used to treat the more difficult U.S. tarry material, should be considered for this application.

Characteristics of the Wastes

The waste products of heavy oil and in-situ bitumen production can be classified as follows:

1. Field Tank Sand accumulates as drop out solids in the small tanks located at or near individual production wells. These sands are periodically collected by trucks and carried to a central collection pit for holding, or for other disposal. Oil content is usually less than 5 percent.
2. Desand Pit Sludge collects from the battery treater operation. The sandy sludge collects over a period of time in the tank or pit, which is periodically cleaned out to permit ongoing

operation. The oil content of the sludge varies widely between 7 percent and 40 percent by weight.

3. Stop Oil and Stop Oil Solids are sand bearing liquids or sludges which usually contain an oil-water emulsion. The emulsion prevents the oil, with its higher density, from separating.
4. Spill Debris is usually soil which has been contaminated with oil from an accidental spill.
5. Ecology Pit Mixture is a composite of the above individual wastes. The ecology pits are central holding facilities which must be periodically emptied to permit ongoing operations to continue. The makeup of the mixture varies widely depending upon the proportion of different types of waste.

The type of materials in the waste from heavy oil and in-situ bitumen production are always the same,

but their proportions can vary greatly. The sand component is very fine, being in the 20 to 200 mesh size range. The oil component is typical of heavy oils, and has a gravity of 12 - 14 deg API. The oil content varies depending upon the waste collection techniques. The wastes usually have a high level of contained salts.

Table 6.2. Estimate of quantities of oily wastes from heavy oil/bitumen operations in 1985.

Location	Quantities (m ³ /yr)	(tonnes/yr)
Cold Lake Area	13,500	17,750
Lindbergh Area	2,500	3,250
Lloydminster Area	87,000	113,000
Total	103,000	134,000

7. Conceptual Design and Cost Analysis of a Mobile Unit for Oily Wastes Clean-up

This chapter includes results of a study on the clean-up of oily wastes from bitumen upgrading plants using the Clean Soil process. A conceptual design and a cost analysis for a mobile plant for clean-up of tar refuse are included in Chapter 8.

7.1. Design Basis and Assumptions

Plant Size and Design

The proposed facility would be a mobile processing unit, skid mounted, which could be easily transported from site to site. A plant design has been developed based on the following assumption:

- Plant capacity would be 6 tonnes/hr, total dry feed to the agglomerator or 4.25 tonnes/hr of oily wastes.

A plant mass balance, based on these assumptions, is given in Table A5.1, Appendix 5. For each stream, water content and percentage of solids are also presented. The plant material balance is based on an oil content of 26 percent by weight in the oily wastes. The oil remaining in the "clean" sand is assumed to be 0.4 percent (w/w). Because of the limited experimental data available on the cleaning of material contaminated by oily wastes, the residence time assumption and equipment size selected are approximate only, and the design and costing should be updated as more data becomes available.

Plant Load Factor

It was assumed that the mobile processing unit will operate on 3 shifts per day, 6 hours per shift, 300 days a year. The plant will process about 30,500 tonnes of oily wastes per year and have a load factor of 62 percent.

Plant Location

It is assumed that the mobile plant operates in the Lloydminster area, processing oily wastes from heavy oil recovery operations.

Plant Equipment

The mobile unit is comprised of an oily waste hopper, a coal hopper, a coal slurry tank, a tumbler washer with trommel screens, a high shear mixer, a three-cell floatation unit as well as agglomerate washing screens, a hydrocyclone and a filter press (Flowsheet #ARC74).

The equipment selected is commercially available.

Environmental Guide

The plant is designed to meet 1989 emission standards in Alberta.

Financial Analysis

All financial analyses in Section 7 were performed in constant 1989 Canadian dollars.

7.2. Plant Description

The conceptual process is shown in Flowsheet #ARC74.

Pulverized coal from the coal hopper is delivered via a screw feeder to a slurry tank where it is slurred with water. The coal slurry is heated with steam from a steam boiler fueled by natural gas.

In a case where pulverized coal is not available on site, a coal pulverizing unit will be added to the process flowsheet. The pulverizing unit is comprised of a primary crusher, a secondary crusher and a grinding mill. Run of mine coal delivered to the site is crushed and pulverized and then stored in the coal hopper for subsequent use.

Oily wastes from the oily wastes hopper is fed through a screw feeder into the tumbler drum to be mixed with the hot coal slurry. The hot coal slurry provides the heat to maintain the mixture in the tumbler at about 185°F. The high solids loading and high shear mixing results in attrition scrubbing of oily material from surfaces of coarse inert solids. The coarse solids are separated by the trommel screens attached to the end of the drum. The top size of the material that enters the high shear mixers was limited to 3 mm. The undersize is passed to floatation cells after agitation in the high shear mixers. The froth from the floatation step is further washed on vibration screens.

The tailings of the floatation step are passed through classifying cyclones to separate materials larger than 500 µm as coarse refuse. The coarse refuse is combined with the rejects from trommel screens and delivered to an open pit near the site for disposal. The underflow from the cyclone is combined with the wash water from agglomerate screens and is sent to the filter press for separation of the fine refuse. The filter cake is collected and sent to the pit area for disposal. The water from the press is recycled to the process.

7.3. Cost Estimates and Financial Analysis

7.3.1. Sources and Methods of Process Cost Estimates

Major equipment was designed and selected according to the mass balance in Table A4.1. Major equipment was then priced using in-house data or cost data in the literature. The two references used for the cost data are:

1. Canadian Institute of Mining and Metallurgy, Special Volume 25, "Mining and Mineral Processing Equipment Costs and Preliminary Capital Cost Estimations," 1982.
2. G.D. Ulrich: "A Guide to Chemical Engineering Process Design and Economics," New York: John Wiley & Sons, 1984.

Purchase prices are updated to first quarter 1989 dollars using either the *M&S Equipment Index for Mining and Milling Industry* or *Chemical Plant Cost Index* reported in *Chemical Engineering*.

7.3.2. Capital and Operating Cost Estimates

A summary of Total Capital Cost for the mobile unit is presented in Table 7.1. The table depicts Direct and Indirect Costs, Owner's Costs and Working Capital.

Table 7.1. Total capital cost - mobile processing plant for treating Lloydminster oily wastes (Scenario 1).

	'000 Cdn\$
Major Equipment Cost including freight	460
Equipment Installation	225
Process Control	147
Piping & Electrical	202
Site & Outside Line	50
Total Direct Cost	1,084
Engineering & Construction Expenses (20% of TDC)	217
Contingency (20% of TDC & Engineering)	260
Total Plant Costs	1,561
Owner's Costs	
Spare parts (5% of TDC)	54
Working Capital (1.5 months of Processing Cost and 1.5 months of Coal Feed Cost)	188
TOTAL CAPITAL COSTS (TCC)	1,803

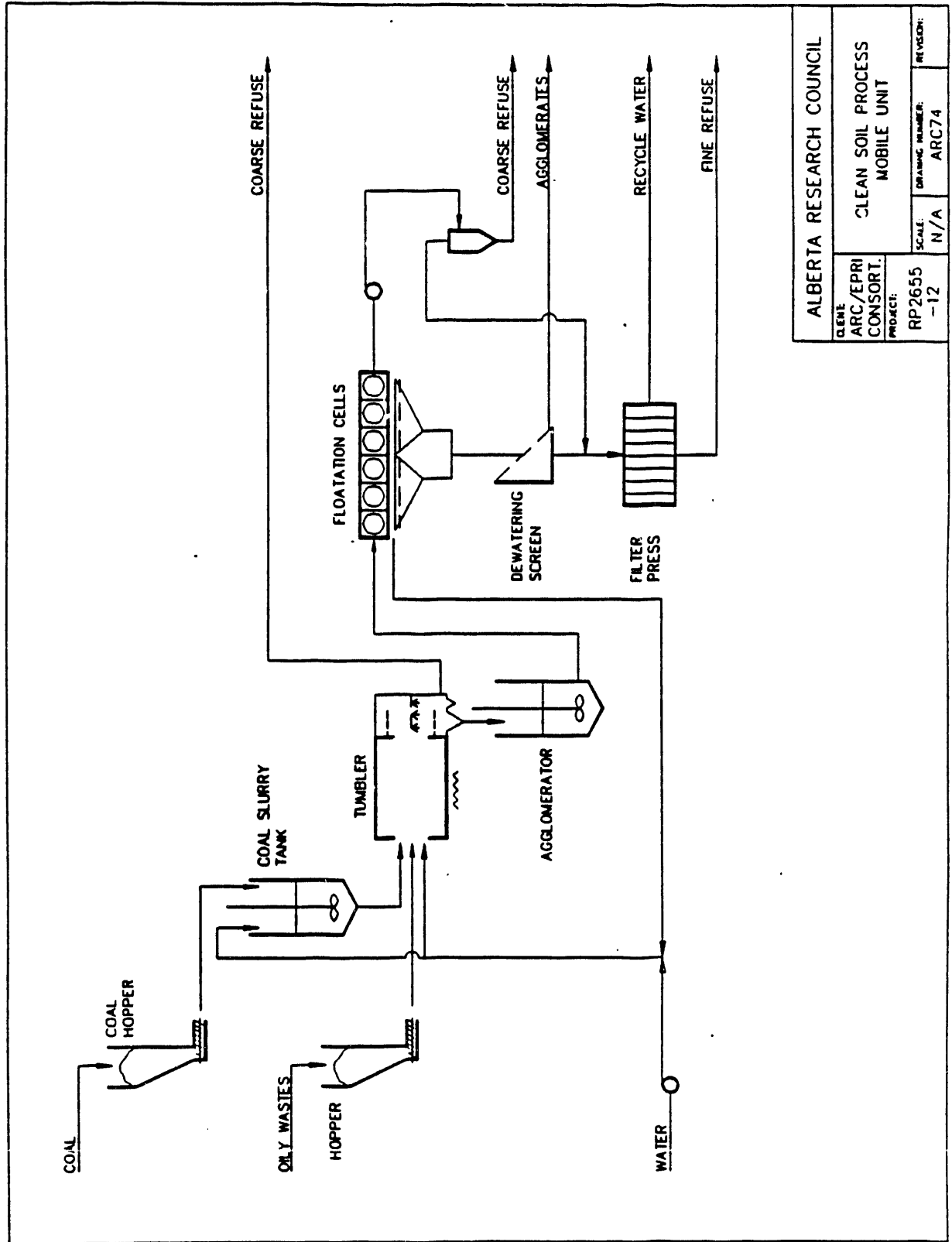
The Home Office and Engineering Costs were estimated at 20 percent of the Total Direct Cost. The Contingency Cost estimates were based on 20 percent of the Direct Costs plus Engineering and Construction Expenses.

A summary of the annual operating costs is presented in Table 7.2. Direct operating costs were estimated as follows:

• coal feed cost	
- run of mine coal	12.00 Cdn\$/tonne
- freight	16.50 Cdn\$/tonne
- total delivered	28.50 Cdn\$/tonne
- pulverized coal delivered	33.50 Cdn\$/tonne
• bridging oil	none added
• electricity	0.04 Cdn\$/kWh
• natural gas	3.00 Cdn\$/mmbtu
• water	no cost, can be recycled
• waste sand disposal	no cost

Table 7.2. Annual operating costs of mobile processing for treating Lloydminster oily wastes (Scenario 1).

	'000 Cdn\$	
Labor		
	Wage Rate ('000\$/Yr)	No/Shift
Foreman	42.00	1
Process Operators	37.00	2
Total		3
Number of Shifts per Day		3
Salaries and Wages		348
Payroll Burden & Overhead (@ 40% of Salaries & Wages)		139
		487
Maintenance & Supplies (@ 5% of TCC)		78
Insurance (@ 1% OF TCC)		16
Natural Gas (0.16 MM GJ/yr @ \$2.00/GJ)		32
Electricity (2.21 MM KWh @ \$0.04/KWh)		88
Total		701
Feedstock		
Pulverized Coal @ \$33.50/tonne		804
Product Credit		
Coal Agglomerates @ 28.50/tonne		-812
NET COST		693



ALBERTA RESEARCH COUNCIL			
CLIENT:	ARC/EPRI	CONSORT.	
PROJECT:	RP2655	-12	
SCALE:	N/A		
DRAWING NUMBER:	ARC74		
REVISION:			

7.3.3. Financial Analysis

The criteria for the financial analysis of the mobile unit included:

- equity financing: 100 percent
- operating life of mobile unit: 15 years
- required real rate of return: 12 percent
- unit load factor: 82 percent
- taxes: not considered

The marketing of the agglomerate product is a concern for the mobile plant operator. The following two scenarios were designed to assess the impact of the two different operating environments.

Scenario 1: Best Case Scenario

This is the case where pulverized coal and agglomerate markets are available at close proximity to the plant site. It represents the case where coal has replaced natural gas in steam raising in heavy oil operations. It is assumed that the agglomerate product can be utilized on site by the heavy oil operator to supplement the coal supply and therefore would fetch a price at least equal to the price of delivered coal. However, no premium is claimed for the higher calorific value of the agglomerate.

Scenario 2: Worst Case Scenario

This is the case where pulverized coal and agglomerate markets are not available at close proximity to the plant site. A coal pulverizing unit must be added to the mobile plant. In addition, the coal agglomerate product has to be shipped out and sold separately. Therefore, freight charges have to be deducted from the selling price. Hence, a lower netback, same as run of mine coal, is assumed.

A summary of the financial analysis for the two scenarios is presented in Table 7.3.

Capital investments for the mobile treatment unit in Scenarios 1 and 2 are estimated at \$Cdn 1.8 million and \$Cdn 2.06 million respectively. The total cost of processing a tonne of the oily waste material ranges from \$Cdn 31.39/tonne in Scenario 1 to \$Cdn 46.97/tonne in Scenario 2. Coal feed cost represents a significant component of the processing cost (\$Cdn 26.36 and \$Cdn 22.43 per tonne respectively). Equally important is operating cost which ranges from \$Cdn 22.98/tonne in Scenario 1 to \$Cdn 25.84/tonne in Scenario 2. The incremental cost is due to pulverizing the coal in Scenario 2. The fourth largest cost element is the capital charge, at \$Cdn 8.68 and \$Cdn 9.92 per tonne respectively.

The economics of the process are influenced by whether the coal agglomerate product can be utilized near the processing site. The difference in coal agglomerate credit for the two scenarios can be as much as \$Cdn 15.42 per tonne of waste processed (26.63 - 11.21). Therefore, the netback value of the coal agglomerate product is a very important factor in determining the economic viability of the process.

Variance Analysis

The sensitivities of capital investment, operating cost, labor cost, and delivered coal prices on the unit processing cost were examined for Scenario 1. The results are shown below.

Base Values (Canadian dollars)

Capital Investments, \$MM	1.80
Delivered Coal Prices, \$/tonne	28.50
Pulverized Coal Price, \$/tonne	33.50
Unit Processing Cost, \$/tonne	31.39

Sensitivities	Change in proc. cost \$Cdn/tonne	Percent change
1. Capital Investment + 25%	+2.17	7.0
2. Operating Cost + 25%	+5.75	18.3
3. Coal Delivered Price + 25%	-1.32	-4.2
4. Labor Cost + 25%	+3.99	12.7
5. Maintenance/Supplies + 25%	+0.64	2.0

Operating Cost is the most sensitive parameter affecting the unit processing cost. The remaining four parameters tested, rank from high to low, as follows:

- Labor Cost
- Capital Investment
- Maintenance/Supplies
- Coal Delivered Price.

Unit processing cost is not sensitive to delivered coal price in Scenario 1. Actually, unit processing cost is reduced by about \$Cdn 1.32/tonne in the case of a 25 percent increase in delivered coal price. This is because the agglomerate product is credited at delivered coal price, and there are more agglomerates produced than feed coal.

7.4. Conclusions

The capital investment for this mobile treatment unit was estimated at \$Cdn 1.80 MM or \$Cdn 2.06 MM including the coal pulverization unit. The cost of

processing a tonne of "typical" Lloydminster oily waste was estimated to range from \$Cdn 31.39/tonne for the best case scenario (Scenario 1 where the coal agglomerates can be utilized near the site) to \$Cdn 46.97/tonne for Scenario 2 where coal agglomerates were to be shipped for sale.

Important factors influencing the economics of the process were:

- the netback price of the coal agglomerates,
- the delivered price of the coal,
- the labor cost, and, to a lesser extent,
- the capital cost.

These are very much dependent on the specific location and infrastructure available.

The current cost of hauling the oily wastes and spreading the wastes on roads is estimated at between \$Cdn 25 - \$Cdn 35/tonne. However, this practice is likely to be considered unacceptable in future operations, and therefore, the estimated processing cost of \$Cdn 30 - \$Cdn 50/tonne is certainly within a range which makes the mobile treatment unit a viable alternative to current practice. Therefore, when the design and costing of the Clean Soil process for the clean-up of oily wastes is updated in the future using more extensive experimental data, the cost of hauling and spreading the wastes on roads should also be updated.

Table 7.3. Costs of processing Lloydminster oily wastes.

	Scenario 1	Scenario 2
Capital Costs (1989 Cdn \$)		
100 tpd Mobile Processing Unit	1,803,000	2,062,000
Coal Feed (tonnes/year)	24,000	24,000
Coal Feed Prices (\$/tonne)	33.50	28.50
Coal Agglomerates Produced (tonnes/year)	28,500	28,500
Coal Agglomerates Prices (\$/tonne)	28.50	12.00
Oily Wastes Process (tonnes/yr)	30,500	30,500
Operating Costs	701,000	788,000
Capital Charges	264,700	302,700
Total Operating & Capital Charges	965,700	1,090,700
Coal Feedstock Costs	804,000	684,000
Agglomerates Credits	-812,250	-342,000
Net Processing Costs	957,450	1,432,700
Per Unit Cost (\$/tonne)	31.39	46.97
Unit Cost Breakdown (\$/tonne)		
Capital	8.68	9.92
Operating	22.98	25.84
Coal	26.36	22.43
Agglomerate Credit	-26.63	-11.21
Total	31.39	46.97

8. Conceptual Design and Cost Analysis of Tar Contaminated Soil Plants

8.1. Introduction

This section reviews the conceptual design and cost estimates for the Clean Soil process when used for cleaning tar contaminated soils. It is based on the Norwest Resources Consultants Inc. report prepared for EPRI in 1989.

The environmental clean-up aspect is a very important feature of the coal agglomeration technology developed by ARC and EPRI. To exploit this feature, a laboratory program was initiated. Process development began on a bench-scale and is now well into the pilot plant stage. In view of the success of the laboratory program, EPRI commissioned Norwest Resources Consultants Inc. to develop a conceptual plant design and prepare preliminary economics for a mobile unit capable of processing 100 tons per day of contaminated soil. The technology developed could be deployed to clean up old gas manufacturing plant sites in the U.S. The findings of the Norwest Report are summarized in this chapter.

Based on ARC's experience working with U.S. tarry material, a logical extension would be to apply the process to the clean-up of oily wastes from heavy oil operations in Western Canada. This is another area where coal agglomeration technology could have a major impact. A conceptual flowsheet was developed and is presented in this chapter. Some preliminary economics are also included in Section 8.2. Note that all costs in Section 8.2 are in constant 1989 U.S. dollars and in U.S. dollars per short tons (\$/t).

8.2. Clean-up Of Manufactured Gas Plant (MGP) Sites

8.2.1. The Problem

Gas manufactured from coal or oil was once an important urban and industrial fuel. It has since been replaced by natural gas. Few manufactured gas plants (MGP) are operating today, but former producing sites are reported to number in the thousands in the U.S. alone. Most are contaminated to some degree by the organic and inorganic wastes of plant operations. They are sitting on valuable land and may present environmental hazards. These sites need to be cleaned and reclaimed before redevelopment can begin.

The task of cleaning up a site is quite involved in terms of material handling. The inorganic contaminants in the soil have to be segregated manually and cleaned separately. However, the organic materials which are primarily tar, could be cleaned using coal agglomeration technology.

The feed material from the MGP sites contains a high concentration of non-extractable carbonaceous matter characterized by wide particle size distribution and a high affinity for tar. In addition, the chemical properties of the tar adversely affect the floatation of the microagglomerates. All these factors make the tarry material a rather difficult material to treat. However, all these difficulties can be handled by modifying the processing steps, for example, by regrinding and reprocessing at the end of a particular step or at the end of the entire process. However, such modification would add to the complexity of the process.

8.2.2. Design Considerations

The tonnage to be cleaned varies from site to site. However, it tends to be small, on the order of 10,000 to 40,000 tons. Therefore, the plant should be built in modules which can be dismantled and transported from one site to another once the clean-up has been completed. The plant must be designed so that it can process a variety of feeds with tar contents ranging from 4 percent to 20 percent. The standard for "clean" soil should meet a target of no more than 0.1 percent by weight of tar (dry basis) in the soil. Moreover, the overall profile of the plant should be kept as low as possible in order to minimize visual impact in urban areas. Finally, the water circuits should be closed to ensure that all potential water-borne pollutants are treated on the site. All vapors should also be collected and treated.

8.2.3. Plant Description

The mobile plant is designed to process 100 tons of contaminated soil per day. The process flowsheet and the material flow streams are shown in Figure 8.1.

- The soil feed is reclaimed from the stockpile by a front-end loader and placed in a hopper equipped with a grizzly to separate +4" size material. The oversize material is rehandled separately. Undersize (-4") material is fed, at a controlled rate of around 5

tph, by conveyor into the tumbler where it joins the coal slurry stream for the initial treatment phase.

Coal sized at 3/8"x0, is loaded from hoppers onto conveyors which take it to a pulverizing mill. The mill reduces the coal to -60 mesh size. A screw feeder then takes the coal from the surge hopper below the mill, either to the slurry preparation tank, or to the high-shear mixer tank upstream of the second stage floatation cells (see below). Coal slurry is prepared in 2 tanks both of which are equipped with heaters to raise the slurry temperature to around 200°F (a temperature of 185°F is required for the material in the tumbler). The ratio of coal/tar is set at 7:1 on a wt/wt basis. The slurry is prepared in 1-hour batches, with the weight of coal determined from samples of the soil to be fed to the plant. Slurry is pumped into the tumbler where it joins the soil feed.

The material leaves the tumbler in 3 streams:

- 4" x 3/8", which is then crushed to -3/8",
- 3/8" x 10 M which joins the crushed 4" x 3/8" material, and
- -10 M.

The 3/8" x 10 M material is conveyed to a concentrating table which separates the lighter gravity coal/tar material from clean soil. The table overflow (coal/tar) goes to a vibro-centrifuge for dewatering and is then conveyed to the coal/tar bin. The table underflow (soil) is screened and is then either crushed to -30 M for further processing by floatation, or if it is clean enough, it is transported to the clean soil conveyor.

The -10 M material from the tumbler is pumped to a screen for accurate separation of the +30 M and -30 M material. The +30 M material joins the feed to the concentrating table while the -30 M material is pumped to the high-shear mixers. From the mixers, at a controlled slurry density of 10 percent, the material passes to two stages of floatation. First stage tailings are re-pulped in a second set of high shear mixers, where additional coal can be added if required. The froth then passes to the second stage floatation cells. As an option, the coarser fraction of the first stage floatation tailings can be reground to liberate mineral matter and to enhance floatation. The reground tailings go to the second set of high shear mixers. Floatation reagents are added as required before each stage.

The combined froth from both stages of floatation is either agglomerated or centrifuged. For agglomeration, the froth is pumped to the low shear

mixers, where oil is added. The coal/tar agglomerates are screened, with screen oversize being conveyed to the coal/tar bin for subsequent loading into trucks. Should the agglomeration phase not be required, the froth from floatation can be dewatered in the vibro-centrifuge, then conveyed to the coal/tar bin.

The tailings from second stage floatation, now containing disposable clean soil, are transported to the thickener via an oversize protection screen. Oversize from the screen (+30 M size) goes to the concentrating table feed. Underflow from the thickener is pumped to a solid bowl centrifuge for dewatering. The centrifuge cake (clean soil) is conveyed to an open stockpile from where it is moved by front-end loaders to the designated disposal site.

Clarified water from the thickener is returned to the dirty water tanks for reuse in the plant. Make-up water is added to the dirty water tanks as required.

Vapor eliminators extract noxious fumes as required from the tumbler, floatation cells and thickener feed slurry.

8.2.4. Costing Basis

- | | |
|---------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| a. Size of Unit: | 100 tpd
Mobile Demonstration Plant |
| b. Base Year: | 1989 U.S. \$ |
| c. Utility Rates: | |
| - Electricity | \$ 0.085/KWh |
| - Oil Binder | \$ 20.00/bbl |
| - Coal | Net zero cost; as revenue from agglomerate (coal plus tar) sales is assumed equal to the cost of feed coal. No credit is claimed for the higher calorific value of the agglomerate. |
| - Steam/Others | Not applicable. |
| d. Operating Labor Rates: | |
| Wages Rate | (U.S. \$/hr) |
| Foreman | 20.00 |
| Control Room Operator | 12.00 |
| Plant Operator | 12.00 |
| Maintenance | 12.00 |
| Lab Technician | 20.00 |
| Loader Operator | 12.00 |
| e. Capitalization: | 15 years on 12 % rate of return; taxes are not considered. |

Table 8.1. Major equipment list.

Description	Equip. #	Weight lbs	HP	Price FOB U.S.\$	Remarks	Supplier
Scalping Screen	SC-01-1	3,000	5	18,000	Vibrax 4x8	Conv. & Equip.
Soil Feed Hopper	BH-01-1	9,333	-	23,333	-	WetCor
Soil Vibro-Feeder	FV-01-1	660	1	8,500	12"W, 60"L	Carrier
Soil Conv. to Tumbler	CV-01-1	2,800	1	9,755	-	Conv. & Equip.
Metal Detector	MD-01-1	600	1	6,000	-	-
Tumbler	TB-01-2	41,900	125	140,360	7'x14'	McLanahan
Soil Conv. to Crusher	CV-02-2	650	0.5	4,000	Type 9C	Hytrol Conveyor
Soil Crusher	CR-01-2	1,500	5	7,775	Jaw Xer, 5"x6"	Sapor
Soil Conv. #1 to Table	CV-03-2	250	1	2,000	Type D-3-16	Clearfield Conv
Tumbler Fines Tank	TK-01-2	600	-	1,500	-	-
Soil/Coal/Tar Screen	SC-03-3	950	2	9,131	MC-481	Syncro-Matic
Oversize Sieve Bend	SC-04-3	900	-	9,500	2'6"rad, 3'wide	Deister HP
Thickener Feed Tank	TK-02-3	1,200	-	2,500	-	-
Screen U/F Tank	TK-03-3	600	-	1,500	-	-
Soil Conveyor	CV-12-3	250	1	2,000	Type D-3-16	Clearfield Conv
Soil Conv. #2 to Table	CV-04-4	3,500	1	12,250	Type D-2-52	Clearfield Conv
Table O/F Tank	TK-04-4	200	-	500	-	-
Concentrating Table	CT-01-4	2,115	1.5	9,900	#14 table	Deister
Table U/F Screen	SC-02-4	225	2	4,387	MC-241	Syncro-Matic
Table U/F Crusher	CR-02-4	3,650	40	27,800	Cage-Paktor 30"	Gundlach
Coal Slurry Tanks (2)	TK-05,06-5	7,708	15	19,284	-	-
Cyclone	CY-01-6	800	-	4,200	D15B-15"dia	Krebs
Cyclone U/F Crusher	CR-03-5	Option	Opt	Option	-	-
Stage 2 High Shear Mixer	TK-07-5	908	1.5	2,284	-	-
High Shear Mixers (3)	TK-08/10-6	2,716	4.5	6,792	-	-
Stg. 1 Cond. Tank	TK-11-6	1,603	-	3,208	-	-
Coal Hopper	BH-02-7	12,000	4	24,000	-	WetCor
Coal Conv. #1 to Mill	CV-05-7	400	7.5	3,200	Type SD-3-26	Clearfield Conv.
Coal Conv. #2 to Mill	CV-06-7	3,920	10	14,292	Type HSDU-52	Clearfield Conv.
Coal Mill	CR-04-8	3,650	40	27,800	Cage-Paktor 30"	Gundlach
Fine Coal Hopper	BH-03-8	750	-	1,500	-	-
Fine Coal Screw Conv. #1	CV-07-8	800	1.5	6,400	Feeds to slurry tanks	-
Fine Coal Screw Conv. #2	CV-08-8	400	0.5	3,200	Feeds to stg. 2 hi-shear mixer	-
Stage 1 Froth Cell	FC-01-9	5,250	40	42,000	2-180 ft ³	Denver
Stage 2 Froth Cell	FC-02-9	5,250	40	42,000	2-180 ft ³	Denver
Froth Tank	TK-12-9	600	-	1,500	-	-
S.B. Centrifuge	CF-01-10	6,300	30	153,000	24"x30"	Bird
Vib-Cent D/W Screen	SC-05-10	950	2	9,131	MC-481	Syncro-Matic
Vibro-Centrifuge	CF-02-10	10,000	50	45,000	EBW	CMI
Low Shear Mixers (2)	TK-13,14-11	4,172	10	10,430	-	-
Dewatering Screen	SC-08-11	950	2	9,131	MC-481	Syncro-Matic
Coal/Tar Conv.	CV-09-12	5,394	3	18,880	-	Conv. & Equip.
Coal/Tar Conv.	CV-11-12	350	3	2,800	Type D-3-26	Clearfield Conv.
Coal/Tar Hopper	BH-04-12	8,381	2	15,050	-	Conv. & Equip.
Thickener	TH-01-13	12,000	5	84,000	Tank in 12 sec.	WetCor
Clean Soil Conveyor	CV-10-14	1,214	1	9,710	-	Conv. & Equip.
Totals		171,327	460	859,441		

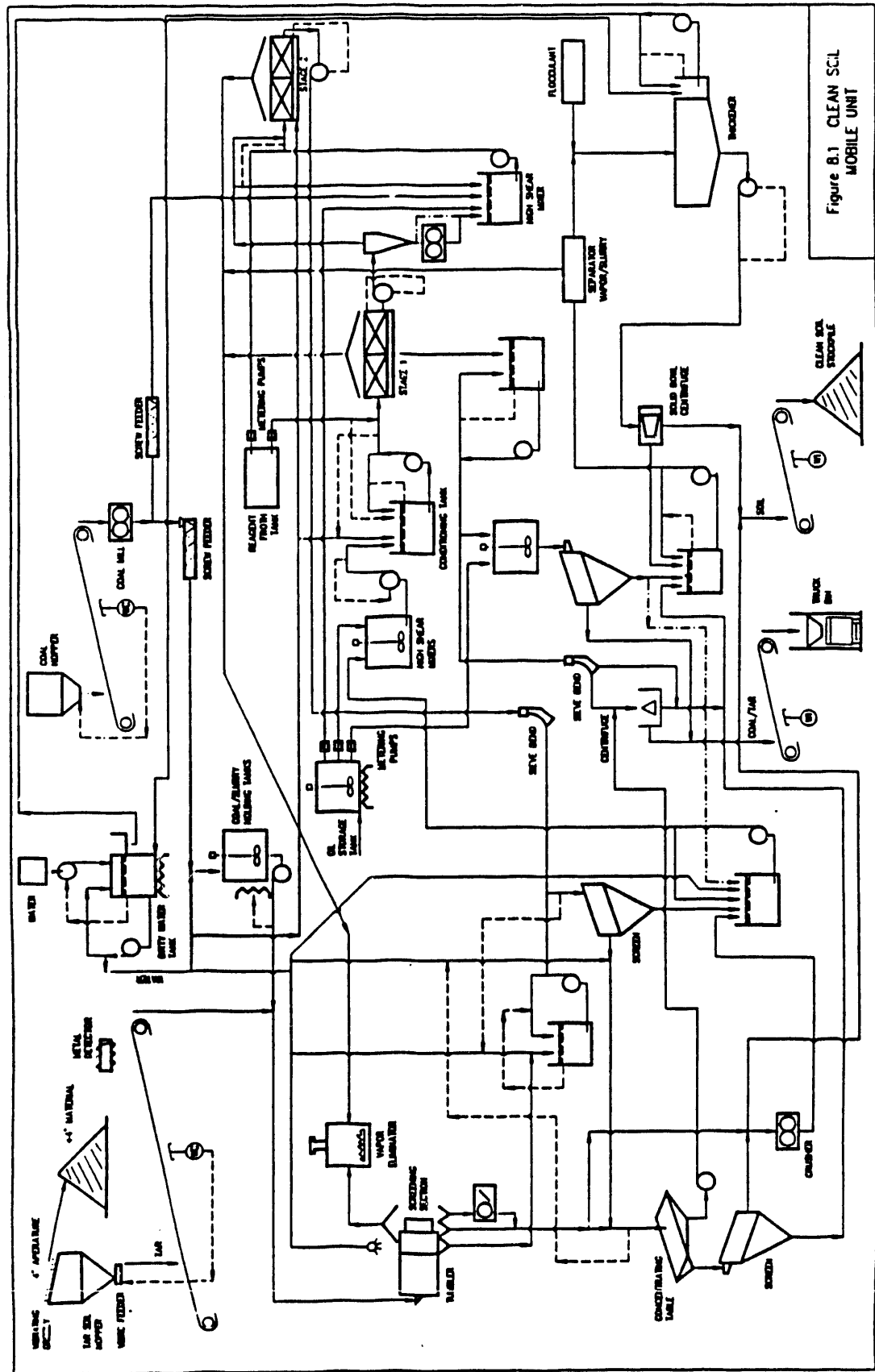


Figure 8.1 CLEAN SCL MOBILE UNIT

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8.3. Economic Evaluation

The presentation of the economics here is somewhat different from the presentation in the Norwest Report. Capitalization cost is included to arrive at a total processing cost. The demonstration plant is amortized for 15 years at a required rate of return of 12 percent. The site installation costs, other than the reuseable building, is expensed rather than capitalized.

The process economics established here are based on a feed with a 20 percent tar content. This represents the worst feed scenario. The economics actually improve with a lower tar content in the feed.

Capital Cost Estimate

A major equipment list, including purchase prices is tabulated in Table 8.1. Capital cost for the 100 tpd demonstration plant is estimated at \$U.S. 2,166,000 (as shown in Table 8.2).

Table 8.2. Capital cost summary: 100 tpd demonstration plant.

	'000 U.S.\$
Main Equipment	859
Freight	49
Pumps, Water Tanks	112
Erection, Piping, Electrical, Instrumentation, etc.	382
Office/Control Room	65
Trailers	141
Total Direct Cost	1,608
Engineering & Construction Expenses	234
Contingency	184
Re-usable Building	140
Total Capital Cost	2,166
Site Installation Costs	81

Table 8.3. Operating cost summary: 100 tpd demonstration plant.

	'000 U.S.\$	\$U.S./ton
Electricity	195	5.35
Reagent	179	4.91
Product Disposal	16	0.43
Labor inc. Overtime	633	22.91
Maintenance & Supplies	148	4.09
Loader Rental	206	5.65
Contingency	158	4.33
Total (excl. oil)	1,735	47.87

Operating Cost Estimate

It is assumed that the plant will operate with 3 8-hour shifts per day. Plant utilization rate is 83 percent. The manpower requirement is estimated as follows:

	No./shift
Foreman	0.33
Control Room Operator	1.00
Plant Operator	1.00
Maintenance Person	1.00
Lab Technician	0.33
Front-end Loader Operator	1.33
Total	5.00

A 5 percent allowance for overtime is to be included. Payroll burden is estimated at 35 percent of salaries and wages.

Total electric power requirement for the plant is about 527 hp. Reagent for the plant is estimated at 71,500 lb/year which is based on a requirement of 1.0 and 0.5 lb/ton of feed for the thickener and floatation units respectively. Cost of reagent is assumed to be \$U.S. 2.50/lb. Lab supplies are estimated at \$U.S. 1,000/month.

Total plant operating cost, excluding oil binder, is presented in Table 8.3. Oil feed is approximately 4 percent by weight of coal feed, or about 0.28 ton/hr.

8.4. Comparison of a 100 tpd Plant vs a 200 tpd Plant

Most of the pieces of equipment selected have capacities significantly higher than the duty envisaged, despite being at the low end of the range of commercial designs. As a result, the plant capacity could be doubled to 200 tpd at an incremental capital cost of about 20 percent. Unit processing cost is reduced from \$U.S. 65.46/ton to about \$U.S. 40.97/ton (Table 8.4). The cost saving occurs be-

Table 8.4. Plant processing cost.

\$/ton	100 TPD	200 TPD
Capital Charges	8.71	5.22
Site Cost	2.22	1.11
Operating	47.87	27.78
Coal	0.00	0.00
Oil	6.86	6.86
Total	65.46	40.97

cause of lower capital charges per unit of wastes processed, and better utilization of labor and of rented loaders. The fixed operating costs such as labor and rental are spread out over a large volume.

8.5. Conclusions and Recommendations

The Norwest Report established the technical feasibility and developed a process flowsheet for a 100 tpd demonstration plant for the treatment of contaminated soil.

1. Capital cost for the 100 tpd plant is estimated at \$U.S. 2,166,000.
2. The cost of processing a ton of tarry wastes is estimated at about \$U.S. 65.46/ton, for feed with a 20 percent tar content.
3. Substantial cost reduction can be achieved with a larger 200 tpd unit.
4. The following process options are identified for further evaluation:
 - regrinding stage 1 tailings;
 - centrifuging coal/tar froth; and
 - gravity separation of 3/8" x 30 mesh material.

9. Summary and Recommendations for Future Work

The objective of this study was to apply advanced agglomeration technology to the cleaning of contaminated soils from manufactured gas plant sites, oil spills, oily waste pits from heavy oil recovery and upgrading plants. The result of the study was the development of the Clean Soil process which, when applied to contaminated soil, yields clean soil suitable for disposal in landfills, and coal agglomerates fuel containing the hydrocarbon contaminants, suitable for combustion in industrial boilers. Specific issues related to the process performance of the Clean Soil process and recommendations for the future work are summarized below.

Batch-Test Studies

Batch-test procedures were developed to provide a rapid screening method for characterization of various contaminated soils for treatment with the Clean Soil process. The tests performed also assisted in improvement of the process and development of process research information required for operation of the Integrated Agglomeration Test Facility (PDU). The parameters identified as important for the cleaning of contaminated tar refuse were:

- conditioning of refuse with a small amount of hydrocarbon solvent,
- intensity of tumbling or agitation with an increased amount of solids being beneficial,
- temperature of tumbling with optimal temperature of 70°C,
- properties of added coal, with bituminous coal showing better performance, and
- addition of froth collectors such as kerosene, etc.

The key properties of contaminated soil refuse were:

- amount and composition of tar,
- presence of coke or other carbonaceous materials, and
- particle size distribution of the soil matrix.

The important parameters identified accounted for all major variations in product properties and process performance. At best conditions, the clean soil product contained less than 0.1 percent of hydrocarbon contaminants. This concentration exceeds current environmental requirements for landfill disposal of soils. However, a few samples were difficult to clean and the clean soil product contained high concentration (0.1 percent) of hydrocarbon contaminants. It is recommended, therefore, that further

detailed batch-tests be conducted to study process research issues such as:

- the effect of selective grinding on release of tar occluded in the pores of coke, char and slag, and
- the effect of contaminant (tar) properties on agglomeration kinetics.

Integrated Agglomeration Test Facility

The IATF runs provided engineering design and operation data for the Clean Soil process. The major process areas were debugged and process control requirements were identified. Difficulties were experienced, however, in obtaining controlled, steady-state performance of the IATF for some feeds. This was due to limited process information on:

- the tumbling mechanisms including the effect of time, solids concentration, temperature and tar/coal ratio;
- the effect of tar distribution in different size fractions on the type of separation (screening) unit required after tumbling;
- the kinetics of agglomeration and the effect of combined oil and solid concentration on micro- and macro-agglomeration;
- the most appropriate approaches to reprocessing of difficult soil samples with various properties; and
- the controllability of the IATF system including measurement of slurry flow, concentration and density.

It is recommended that all these issues be studied and further developed.

Product Testing and Characterization

The product agglomerates from the IATF were tested for handling, grinding and combustion characteristics and the results were compared to the test burns with the parent HVB coal. The tests showed agglomerates to be suitable for combustion in existing industrial boilers, and the combustion emissions to be similar to the emissions for HVB coal used in agglomeration. However, because only one agglomerate product was evaluated, recommendations were made for further testing, namely:

- Test samples using different coal and contaminant hydrocarbons should be evaluated in terms of their combustion emissions and acceptability of the ash for disposal.
- Larger test samples should be evaluated for handling, flowability, combustion, stockpile stability, etc.

- The effect of process parameters and product properties such as moisture, oil content, etc. on compaction of agglomerates should be further studied.

Feasibility of the Clean Soil Process

A conceptual design for the Clean Soil process was developed for treatment of oily wastes from bitumen upgrading. The processing unit was assumed to have a load factor of 82 percent and to process about 30,500 tonnes of oily wastes per year.

The cost of processing a tonne of oily wastes was estimated to range from \$Cdn 31.39/tonne for a scenario where markets for the agglomerates byproduct exist close to the mobile plant site, to \$Cdn 46.97/tonne for a scenario where agglomerates have to be shipped for sale. These costs compared favorably with the estimated cost for haul-

ing and spreading the wastes on roads (\$Cdn 25 to \$Cdn 35/tonne). In view of the fact that spreading oily wastes on roads may not be acceptable in the future, the Clean Soil process offers a viable, alternative solution to oil waste cleanup in the bitumen and heavy oil upgrading industry.

Further recommended studies on the feasibility of the process are:

- upgrading of the design and costing of the process based on additional experimental work with oily wastes;
- updating of the process economics based on site specific application of the mobile plant in the Lloydminster area; and
- review of the costs of current and alternate practices for oily wastes disposal and comparison of these costs to the updated cost of clean-up using the Clean Soil process.

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Appendix 2.

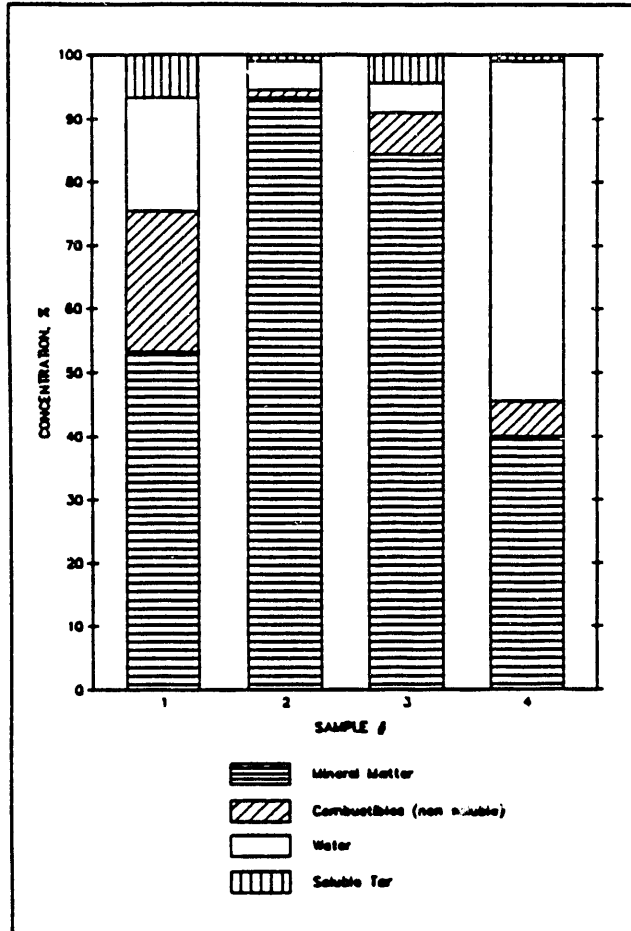


Figure A2.1. Composition of tar refuse samples (Site #1).

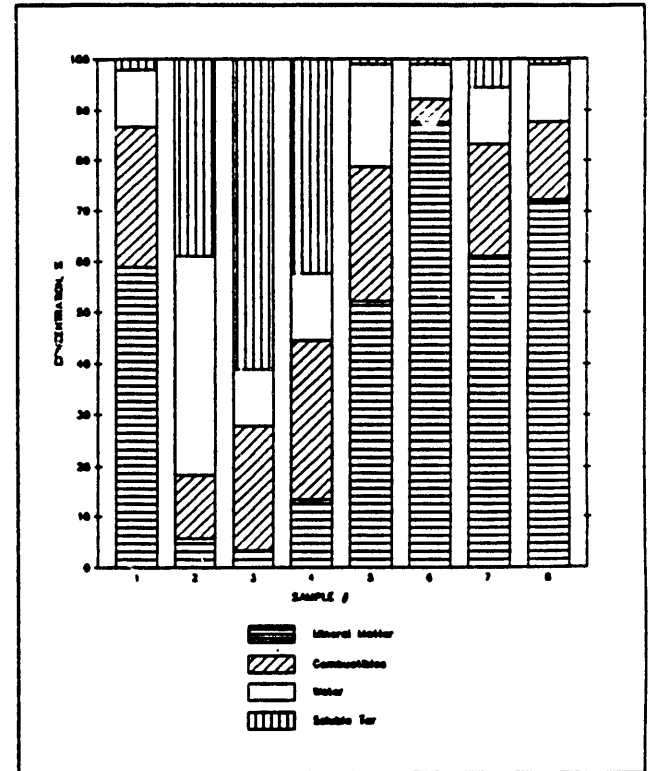


Figure A2.2. Composition of tar refuse samples (Site #2).

Table A2.4. Site #1-1: Clean-up of contaminated soil - overall material balance (%)^a.

Streams	Assays Distribution			Mineral matter/ash	Tar/oil	Combust	Mineral matter/ash
	Weights	tar/oil	Combust				
Contaminated Soil	71.88	5.53	9.16	85.31	64.7	21.7	96.5
Coal	25.95	-	91.31	8.69	-	78.3	3.5
Froth Collector	2.17	100.00	-	-	35.3	-	-
Total Feed	100.00	6.14	30.28	83.57	100.0	100.0	100.0
Total Product	98.45	5.01	30.52	62.92	81.5	100.8	99.0
Combustible Product	60.18	8.21	49.83	41.93	80.5	99.1	39.7
Clean Soil	32.60	0.07	0.70	99.23	0.4	0.7	50.9
Reject	5.67	0.69	5.16	94.15	0.6	1.0	8.4

a: on moisture free basis.

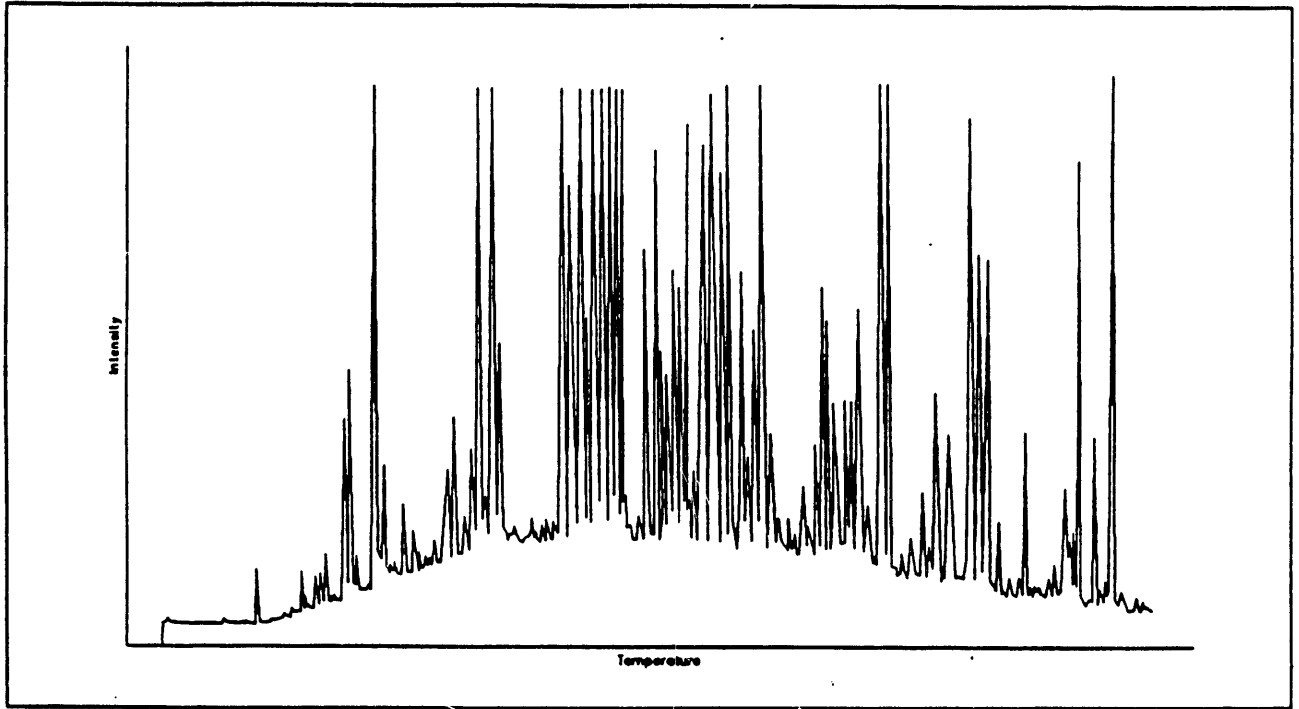


Figure A2.3. Capillary gas chromatogram of tar (Sample 1).

RADIAN ANALYSIS
3/16/89
SITE 1-1 FEED
UNITS UG/G

EPA METHOD 8240 VOLATILES

BENZENE	.66
ETHYLBENZENE	14.00
STYRENE	9.4
TOLUENE	16.00
TOTAL XYLENES	34.00

SUBTOTAL 74.06

EPA METHOD 8270 SEMIVOLATILES

ACENAPHTHYLENE	110.00
FLUORANTHENE	76.00
FLUORENE	57.00
2-METHYLNAPHTHALENE	270.00
NAPHTHALENE	110.00
PHENANTHRENE	310.00
PYRENE	110.00
UNKNOWN	731.00
DIMETHYLCYCLOHEXANE	73.00
ETHYLBENZENE	350.00
C10H10	22.00
1,1'-BIPHENYL	34.00
DIMETHYLNAPHTHALENE	60.00
DIMETHYLNAPHTHALENE	77.00
DIMETHYLNAPHTHALENE	33.00
C12H10	32.00
ETHYLNAPHTHALENE	35.00
METHYLETHYLNAPHTHALENE	20.00
1H-PHENALENE	22.00
DIBENZOTHIOPHENE	21.00
METHYLANTHRACENE	39.00
METHYLANTHRACENE	28.00
C15H12	22.00
METHYLPYRENE	25.00

SUBTOTAL 2567.00
2641.06

RADIAN ANALYSIS
3/16/89
SITE 1-1 FROTH
UNITS UG/G

EPA METHOD 8240 VOLATILES

BENZENE	2.3
CHLOROFORM	.25
ETHYLBENZENE	1.20
METHYLENE CHLORIDE	1.80
STYRENE	4.70
TOLUENE	5.70
TOTAL XYLENES	4.80

SUBTOTAL 18.45

EPA METHOD 8270 SEMIVOLATILES

ACENAPHTHALENE	380.00
ANTHRACENE	230.00
BENZO(A)ANTHRACENE	200.00
BENZO(A)PYRENE	150.00
BENZO(B)FLUORANTHENE	180.00
BENZO(GHI)PERYLENE	77.00
BENZO(K)FLUORANTHENE	180.00
CHRYSENE	180.00
DIBENZOFURAN	56.00
FLUORENE	220.00
INDENO(1,2,3-CD)PYRENE	60.00
2-METHYLNAPHTHALENE	350.00
NAPHTHALENE	140.00
PHENANTHRENE	1000.00
PYRENE	700.00
UNKNOWN	1503.00
ETHYLBENZENE	330.00
DIMETHYLNAPHTHALENE	140.00
DIMETHYLNAPHTHALENE	200.00
DIHYDROACENAPHTHYLENE	83.00
DIMETHYLNAPHTHALENE	94.00
METHYLFLUORENE	57.00
DIBENZOTHIOPHENE	75.00
C15H12	120.00
METHYLPHENANTHRENE	120.00
METHYLPHENANTHRENE	80.00
ETHYLANTHRACENE	54.00
METHYLPYRENE	180.00

SUBTOTAL 7139.00

TOTAL 157.45

RADIAN ANALYSIS
3/23/89
SITE 1-1 TAILINGS
UNITS UG/G

EPA METHOD 8240 VOLATILES

STYRENE	2.20
SUBTOTAL	2.20

EPA METHOD 8270 SEMIVOLATILES

ACENAPHTHALENE	6.50
ACENAPHTHENE	.82
ANTHRACENE	7.60
BENZO(A)ANTHRACENE	8.90
BENZO(B)FLUORANTHENE	9.00
BENZO(GHI)PERYLENE	4.70
BENZO(K)FLUORANTHENE	9.80
BENZYL ALCOHOL	2.50
CHRYSENE	9.10
O-CRESOL	.63
DIBENZO(A,H)ANTHRACENE	1.60
DIBENZO(B,K)ANTHRACENE	1.50
FLUORANTHENE	18.00
FLUORENE	2.50
INDENO(1,2,3-CD)PYRENE	3.60
2-METHYLNAPHTHALENE	5.00
NAPHTHALENE	1.90
PHENANTHRENE	42.00
PYRENE	26.00
ETHYLBENZENE	7.40
DIMETHYLBENZENE	5.10
DIMETHYLNAPHTHALENE	2.10
DIMETHYLNAPHTHALENE	2.90
TRIDECANE	0.87
METHYLFLUORENE	0.71
UNKOWNS	1.78
C15H12	1.60
METHYLPHENANTHRENE	1.40
C15H10	2.30
C15H12	1.10
C16H12	1.00
DIMETHYLPHENANTHRENE	1.10
METHYLPYRENE	1.90
C17H12	1.10
C17H12	0.79
METHYLPYRENE	0.80
METHYLPYRENE	0.66

SUBTOTAL	137.26
TOTAL	139.46

RADIAN ANALYSIS
3/23/89
SITE 1-1 PROCESS WATER
UNITS UG/L

EPA METHOD 8240 VOLATILES

BENZENE	18.00
ETHYLBENZENE	3.20
4-METHYL-2-PENTANONE	560.00
STYRENE	7.70
TOLUENE	26.00
TOTAL XYLENES	12.00

SUBTOTAL	826.36
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EPA METHOD 8270 SEMIVOLATILES

ACETOPHENONE	38.00
2,4-DIMETHYLPHENOL	96.00
UNKOWNS	1534.00
DIMETHYLPHENOL	100.00
BENZO(B)THIOPHENE	130.00
C9H8O	160.00
C8H8O2	36.00
DIHYDROINDENONE	870.00
ISOBENZOFURANONE	75.00
C12H10	97.00
NAPHTHALENECARBOXYLIC ACID	44.00
NAPHTHOPYRANONE	140.00

SUBTOTAL	3320.00
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TOTAL	4146.36
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RADIAN ANALYSIS
3/29/89
SITE 1-1
UNITS UG/ML

METALS ANALYSES

	FEED	FROTH	TAILINGS	WATER
AG	<0.03	<0.03	<0.03	<0.03
AS	0.003	0.004	<0.002	<0.002
BA	0.25	0.35	0.10	0.051
CD	<0.005	<0.005	<0.005	<0.005
CR	<0.03	<0.03	<0.03	<0.03
PB	0.1	<0.05	0.1	<0.05
SE	<0.002	<0.002	0.005	<0.002
HG		<0.0002	<0.0002	<0.0002

Analytical Data Summary

EPRI

Radian Work Order: 89-07-121

Method:SU8240-Volatile Organics (1)

List:

Sample ID:	SITE #2-2 FEED	SITE #2-2 FRO- TH	SITE #2-2 TAI- LINGS	TCLP BLANK	SYSTEM BLANK
Factor:	10.000	5.000	1.000	1.000	1.00
Results in:	ug/L	ug/L	ug/L	ug/L	ug/L
	010	020	030	05A	07A
Matrix:	leachate	leachate	leachate	leachate	leachate
Acrylonitrile	<250	<130	<25	<25	<25
Benzene	<50	<25	<5.0	<5.0	<5.0
Carbon disulfide	<50	<25	<5.0	<5.0	<5.0
Carbon tetrachloride	<50	<25	<5.0	<5.0	<5.0
Chlorobenzene	<50	<25	<5.0	<5.0	<5.0
Chloroform	<50	<25	<5.0	<5.0	<5.0
1,2-Dichloroethane	<50	<25	<5.0	<5.0	<5.0
1,1-Dichloroethene	<50	<25	<5.0	<5.0	<5.0
Methyl ethyl ketone	<1000	<500	<100	<100	<100
Methylene chloride	<50	<25	<5.0	<5.0	<5.0
1,1,1,2-Tetrachloroethane	<50	<25	<5.0	<5.0	<5.0
1,1,2,2-Tetrachloroethane	<50	<25	<5.0	<5.0	<5.0
Tetrachloroethene	<50	<25	<5.0	<5.0	<5.0
Toluene	<50	<25	<5.0	<5.0	<5.0
1,1,1-Trichloroethane	<50	<25	<5.0	<5.0	<5.0
1,1,2-Trichloroethane	<50	<25	<5.0	<5.0	<5.0
Trichloroethene	<50	<25	<5.0	<5.0	<5.0
Vinyl chloride	<100	<50	<10	<10	<10

Surrogate Recovery(%)

1,4-Bromofluorobenzene	91	91	93	88	91
Control Limits: 55 to 167					
1,2-Dichloroethane-d4	94	95	97	98	97
Control Limits: 39 to 156					
Toluene-d8	99	98	100	97	103
Control Limits: 58 to 146					

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

Analytical Data Summary

EPRI

Radian Work Order: 89-07-121

Method: SUB270-Semi-Volatiles (1)

List:

Sample ID:	SITE #2-2 FEED	SITE #2-2 FROTH	SITE #2-2 TAILINGS	REAGENT BLANK	TAILINGS REAGENT BLANK
Factor:	4.76	4.93	0.1	1.0	0.1
Results in:	ug/g	ug/g	ug/g	ug/g	ug/g
Matrix:	FEED	FROTH	TAILINGS	BLANK	BLANK
Acenaphthene	<u>120*</u>	<u>95*</u>	<u>3.0*</u>	<10	<1.0
Acenaphthylene	<u>340</u>	<u>270</u>	<u>5.7</u>	<10	<1.0
Acetophenone	<48	<49	<1.0	<10	<1.0
4-Aminobiphenyl	<48	<49	<1.0	<10	<1.0
Aniline	<48	<49	<1.0	<10	<1.0
Anthracene	<u>230*</u>	<u>160*</u>	<u>5.5</u>	<10	<1.0
Benzidine	<48	<49	<1.0	<10	<1.0
Benzo(a)anthracene	<u>200*</u>	<u>170*</u>	<u>8.0</u>	<10	<1.0
Benzo(a)pyrene	<u>160*</u>	<u>130*</u>	<u>6.3</u>	<10	<1.0
Benzo(b)fluoranthene	<u>96*</u>	<u>76*</u>	<u>4.7*</u>	<10	<1.0
Benzo(g,h,i)perylene	<u>59*</u>	<49	<u>2.1*</u>	<10	<1.0
Benzo(k)fluoranthene	<u>71*</u>	<u>87*</u>	<u>4.5*</u>	<10	<1.0
Benzoic acid	<48	<49	<1.0	<10	<1.0
Benzyl alcohol	<95	<99	<2.0	<20	<u>67</u>
4-Bromophenyl phenyl ether	<48	<49	<1.0	<10	<1.0
Butylbenzylphthalate	<48	<49	<1.0	<10	<1.0
4-Chloro-3-methylphenol	<95	<99	<2.0	<20	<2.0
p-Chloroaniline	<95	<99	<2.0	<20	<2.0
Chlorobenzilate	<48	<49	<1.0	<10	<1.0
bis(2-Chloroethoxy)methane	<48	<49	<1.0	<10	<1.0
bis(2-Chloroethyl)ether	<48	<49	<1.0	<10	<1.0
bis(2-Chloroisopropyl)ether	<48	<49	<1.0	<10	<1.0
1-Chloronaphthalene	<48	<49	<1.0	<10	<1.0
2-Chloronaphthalene	<48	<49	<1.0	<10	<1.0
2-Chlorophenol	<48	<49	<1.0	<10	<1.0
4-Chlorophenyl phenyl ether	<48	<49	<1.0	<10	<1.0
Chrysene	<u>250</u>	<u>200*</u>	<u>10</u>	<10	<1.0
Di-n-octylphthalate	<48	<49	<1.0	<10	<1.0
Dibenz(a,h)anthracene	<48	<49	<1.0	<10	<1.0
Dibenz(a,j)acridine	<48	<49	<1.0	<10	<1.0

* Est. result less than 5 times detection limit

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

Analytical Data Summary

EPRI

Radian Work Order: 89-07-121

Method: SW8270-Semi-Volatiles (1)

List:

Sample ID:	SITE #2-2 FEED	SITE #2-2 FRO- TH	SITE #2-2 TAI- LINGS	REAGENT BLANK	TAILINGS REAG- ENT BLANK
Factor:	4.76	4.93	0.1	1.0	0.1
Results in:	ug/g	ug/g	ug/g	ug/g	ug/g
Matrix:	FEED	FROTH	TAILINGS	BLANK	BLANK
Dibenzofuran	<48	<49	<1.0	<10	<1.0
Dibutylphthalate	<48	<49	<1.0	<10	<1.0
1,2-Dichlorobenzene	<48	<49	<1.0	<10	<1.0
1,3-Dichlorobenzene	<48	<49	<1.0	<10	<1.0
1,4-Dichlorobenzene	<48	<49	<1.0	<10	<1.0
3,3'-Dichlorobenzidine	<95	<99	<2.0	<20	<2.0
2,4-Dichlorophenol	<48	<49	<1.0	<10	<1.0
2,6-Dichlorophenol	<48	<49	<1.0	<10	<1.0
Diethylphthalate	<48	<49	<1.0	<10	<1.0
p-Dimethylaminoazobenzene	<48	<49	<1.0	<10	<1.0
7,12-Dimethylbenz(a)anthracene	<95	<99	<2.0	<20	<2.0
Dimethylphenethylamine	<48	<49	<1.0	<10	<1.0
2,4-Dimethylphenol	<48	<49	<1.0	<10	<1.0
Dimethylphthalate	<48	<49	<1.0	<10	<1.0
4,6-Dinitro-2-methylphenol	<240	<250	<5.0	<50	<5.0
1,3-Dinitrobenzene	<48	<49	<1.0	<10	<1.0
2,4-Dinitrophenol	<240	<250	<5.0	<50	<5.0
2,4-Dinitrotoluene	<48	<49	<1.0	<10	<1.0
2,6-Dinitrotoluene	<48	<49	<1.0	<10	<1.0
Diphenylamine	<48	<49	<1.0	<10	<1.0
1,2-Diphenylhydrazine	<48	<49	<1.0	<10	<1.0
Ethyl methanesulfonate	<48	<49	<1.0	<10	<1.0
bis(2-Ethylhexyl)phthalate	<48	<49	8.0	<10	<1.0
Fluoranthene	610	350	12	<10	<1.0
Fluorene	280	310	8.4	<10	<1.0
Hexachlorobenzene	<48	<49	<1.0	<10	<1.0
Hexachlorobutadiene	<48	<49	<1.0	<10	<1.0
Hexachlorocyclopentadiene	<48	<49	<1.0	<10	<1.0
Hexachloroethane	<48	<49	<1.0	<10	<1.0
Indeno(1,2,3-cd)pyrene	60*	<49	2.2*	<10	<1.0

* Est. result less than 5 times detection limit

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

Analytical Data Summary

EPRI

Radian Work Order: 89-07-121

Method: SW8270-Semi-volatiles (1)

List:

Sample ID:	SITE #2-2 FEED	SITE #2-2 FROTH	SITE #2-2 TAILINGS	REAGENT BLANK	TAILINGS REAGENT BLANK
Factor:	4.76	4.93	0.1	1.0	0.1
Results in:	ug/g	ug/g	ug/g	ug/g	ug/g
Matrix:	FEED	FROTH	TAILINGS	BLANK	BLANK
Isophorone	<48	<49	<1.0	<10	<1.0
Methyl methanesulfonate	<48	<49	<1.0	<10	<1.0
3-Methylcholanthrene	<48	<49	<1.0	<10	<1.0
2-Methylnaphthalene	<u>770</u>	<u>670</u>	<1.0	<10	<1.0
2-Methylphenol(o-cresol)	<48	<49	<u>1.2*</u>	<10	<1.0
4-Methylphenol(p-cresol)	<48	<49	<1.0	<10	<1.0
N-Nitroso-di-n-butylamine	<48	<49	<1.0	<10	<1.0
N-Nitrosodimethylamine	<48	<49	<1.0	<10	<1.0
N-Nitrosodiphenylamine	<48	<49	<1.0	<10	<1.0
N-Nitrosodipropylamine	<48	<49	<1.0	<10	<1.0
N-Nitrosopiperidine	<48	<49	<1.0	<10	<1.0
Naphthalene	<u>310</u>	<u>360</u>	<u>7.5</u>	<10	<1.0
1-Naphthylamine	<48	<49	<1.0	<10	<1.0
2-Naphthylamine	<48	<49	<1.0	<10	<1.0
5-Nitro-o-toluidine	<48	<49	<1.0	<10	<1.0
2-Nitroaniline	<240	<250	<5.0	<50	<5.0
3-Nitroaniline	<240	<250	<5.0	<50	<5.0
4-Nitroaniline	<240	<250	<5.0	<50	<5.0
Nitrobenzene	<48	<49	<1.0	<10	<1.0
2-Nitrophenol	<95	<99	<2.0	<20	<2.0
4-Nitrophenol	<240	<250	<5.0	<50	<5.0
Pentachlorobenzene	<48	<49	<1.0	<10	<1.0
Pentachloronitrobenzene	<48	<49	<1.0	<10	<1.0
Pentachlorophenol	<240	<250	<5.0	<50	<5.0
Phenacetin	<48	<49	<1.0	<10	<1.0
Phenanthrene	<u>1200</u>	<u>860</u>	<u>68</u>	<10	<1.0
Phenol	<48	<49	<1.0	<10	<1.0
2-Picoline	<48	<49	<1.0	<10	<1.0
Pronamide	<48	<49	<1.0	<10	<1.0
Pyrene	<u>550</u>	<u>690</u>	<u>16</u>	<10	<1.0

* Est. result less than 5 times detection limit

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

Analytical Data Summary

EPRI

Radian Work Order: 89-07-121

Method:SV8270-Semi-volatiles (1)

List:

Sample ID:	SITE #2-2 FEED	SITE #2-2 FROTH	SITE #2-2 TAILINGS	REAGENT BLANK	TAILINGS REAGENT BLANK
Factor:	4.76	4.93	0.1	1.0	0.1
Results in:	ug/g	ug/g	ug/g	ug/g	ug/g
Matrix:	FEED	FROTH	TAILINGS	BLANK	BLANK
1,2,4,5-Tetrachlorobenzene	<48	<49	<1.0	<10	<1.0
2,3,6-Tetrachlorophenol	<95	<99	<2.0	<20	<2.0
1,2,4-Trichlorobenzene	<48	<49	<1.0	<10	<1.0
2,4,5-Trichlorophenol	<48	<49	<1.0	<10	<1.0
2,4,6-Trichlorophenol	<48	<49	<1.0	<10	<1.0

Surrogate Recovery(%)

2-Fluorophenol	95	89	NC	103	NC
Control Limits: 20 to 158					
Phenol-d5	75	72	25 0	80	19 0
Control Limits: 27 to 154					
Nitrobenzene-d5	82	76	44	81	49
Control Limits: 21 to 159					
2-Fluorobiphenyl	109	105	91	101	80
Control Limits: 33 to 153					
2,4,6-Tribromophenol	82	77	57	42	35
Control Limits: 0 to 179					
Terphenyl-d14	43	57	110	47	45
Control Limits: 0 to 223					
Acridine-d9	NS	NS	66	NS	72
Control Limits: to					

NS Not spiked

NC Not calculated

0 Outside control limits

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

Appendix 3.

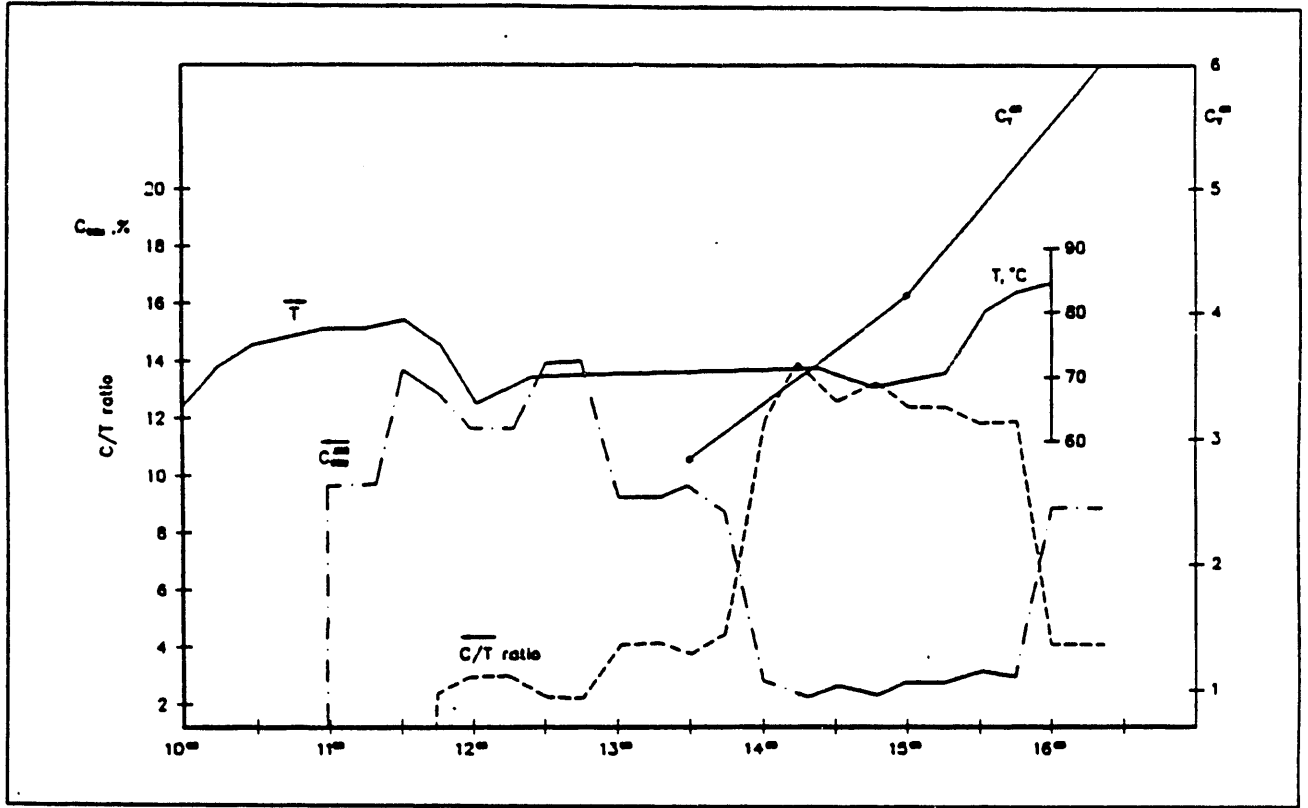


Figure A3.1. IATF Run No. 6 (Process Variables).

Appendix 4.

Table A4.1. Polynuclear aromatic hydrocarbons measured from the 100% Site 2 coal and MGP 2 blend combustion tests.

analyte	25% MGPW Blend		100% Site 2 coal	
	Test Run 1	Test Run 2	Test Run 1	Test Run 2
Napthalene	ND	ND	ND	ND
Acenaphthylene	ND	ND	ND	ND
Acenaphthene	ND	ND	ND	ND
Fluorene	ND	ND	ND	ND
Phenanthrene	ND	ND	ND	ND
Anthracene	ND	ND	ND	ND
Fluoranthene	ND	ND	ND	ND
Pyrene	ND	ND	ND	ND
Pyrene	ND	ND	ND	ND
Benz (a) anthracene	ND	ND	ND	ND
Chrysene	ND	ND	ND	ND
Benzo (b) fluoanthene	ND	ND	ND	ND
Benzo (k) fluoant:ene	ND	ND	ND	ND
Benzo (a) pyrene	ND	ND	ND	ND
Dibenz (a,h) anthracene	ND	ND	ND	ND
Benzo (g,h,i) perylene	ND	ND	ND	ND
Ideno (1,2,3-cd) pyrene	ND	ND	ND	ND

Method: NIOSH 5515
 ND - non-detectable
 LOD - 1.0 mg

Table A4.2. Volatile organic compounds measured from the 100% Site 2 coal and MGP2 blend combustion tests.

Compound	25% MGP2 blend						100% Site 2 coal					
	$\mu\text{g}/\text{m}^3$	$\mu\text{g}/\text{m}^3$	lb/hr	$\mu\text{g}/\text{m}^3$	$\mu\text{g}/\text{m}^3$	lb/hr	$\mu\text{g}/\text{m}^3$	$\mu\text{g}/\text{m}^3$	lb/hr	$\mu\text{g}/\text{m}^3$	$\mu\text{g}/\text{m}^3$	lb/hr
Benzene	5.7	BDL	-	9.0	2.1	1.35E-5	8.1	BDL	-	3.0	BDL	-
Toluene	52.2	8.2	2.6E-5	95.2	66.7	4.28E-4	67.6	31.1	2.00E-4	66.0	33.6	2.46E-4
Xylenes	1.6	BDL	-	28.0	23.3	1.49E-4	ND	ND	-	ND	ND	-
Ethylbenzene	ND	ND	-	14.8	14.8	9.52E-5	ND	ND	-	1.8	1.8	1.31E-5
Methylcyclohexane	1.6	1.6	5.08E-6	ND	ND	-	2.0	2.0	1.29E-5	ND	ND	-
Tricyclodecane	ND	ND	-	2.1	2.1	1.35E-5	ND	ND	-	ND	ND	-
Trichlorofluoromethane	ND	ND	-	ND	ND	-	4.7	BDL	-	ND	ND	-
1,1,2-Trichloro-1,2,2-trifluoroethane	ND	ND	-	ND	ND	-	2.7	BDL	-	ND	ND	-
Tetrachloroethene	15.5	BDL	-	21.7	10.6	6.82E-5	19.6	5.4	3.47E-5	17.4	4.8	3.52E-5
Chlorobenzene	0.8	0.8	2.53E-6	ND	ND	-	ND	ND	-	ND	ND	-
Hexane Nitrile	ND	ND	-	ND	ND	-	2.7	2.7	1.74E-5	ND	ND	-
Heptane Nitrile	ND	ND	-	0.5	0.5	3.21E-6	2.0	2.0	1.29E-5	6.0	6.0	4.40E-5
Octane Nitrile	ND	ND	-	ND	ND	-	2.0	2.0	1.29E-5	ND	ND	-
Benzo Nitrile	ND	ND	-	0.5	0.5	3.21E-6	ND	ND	-	ND	ND	-
2-Furan-carboxaldehyde	ND	ND	-	1.1	1.1	7.08E-6	ND	ND	-	ND	ND	-
Benzaldehyde	1.6	1.6	5.08E-6	15.3	15.3	9.84E-5	1.3	1.3	8.37E-6	2.4	2.4	1.76E-5
Acetic Acid	ND	ND	-	63.5	63.5	4.08E-4	ND	ND	-	ND	ND	-
Substituted Decanoic Acid	ND	ND	-	ND	ND	-	1.3	BDL	-	ND	ND	-
Hydroxybutanedioic Acid	ND	ND	-	ND	ND	-	ND	ND	-	1.2	1.2	8.78E-6
Heptane	ND	ND	-	3.7	3.7	2.38E-5	ND	ND	-	ND	ND	-
5-methylisoxazole	ND	ND	-	ND	ND	-	0.7	0.7	4.51E-5	ND	ND	-
Phthalic anhydride	0.8	0.8	2.54E-6	ND	ND	-	ND	ND	-	ND	ND	-
Hexamethylcyclotrisiloxane	5.7	BDL	-	ND	ND	-	101.3	47.3	3.0E-6	294.3	246.2	1.76E-3
Hexamethyltrisiloxane	ND	ND	-	ND	ND	-	20.9	18.2	1.17E-4	120.1	117.7	8.61E-4

* = field blank corrected

ND = not detected

BDL = blank correction below detectable limits.

Appendix 5.

Table A5.1. Coal Agglomeration process for cleaning typical Lloydminster sludge.

	Material balance (kg/hr)				Water	Stream total	% solids & tar
	Tar	Coal/ coke	Inert solids	Total dry			
1	1,000.0	-	2,202.0	3,302.0	932.0	4,234.0	77.99
2	-	2,517.0	380.0	2,897.0	434.0	3,331.0	86.97
3a	-	-	-	0.0	5,975.0	5,975.0	0.00
3b	-	-	-	0.0	6,410.2	6,410.2	0.00
3c	-	-	-	0.0	82,141.2	82,141.2	0.00
3d	-	-	-	0.0	6,000.0	6,000.0	0.00
4	-	2,517.0	380.0	2,897.0	6,409.0	9,306.0	31.13
5	0.1	-	250.0	250.1	17.5	267.6	93.48
6	1,099.9	2,517.0	2,332.0	5,948.9	13,733.5	19,682.4	30.22
7	1,097.1	2,459.9	930.5	4,487.5	5,084.2	9,571.7	48.88
8	2.2	67.0	468.9	538.1	9,060.5	9,578.6	5.59
9	2.8	57.1	1,401.5	1,461.4	90,790.5	92,251.9	1.58
10	1,094.9	2,392.9	463.6	3,951.4	2,023.7	5,975.1	68.13
11	2.5	72.7	607.1	682.3	99,184.0	99,866.3	0.68
12	2.5	72.7	607.1	682.3	462.4	1,144.7	59.61
13	0.0	0.0	0.0	0.0	98,721.6	98,721.6	0.00
14	2.5	51.4	1,261.3	1,315.2	667.0	1,982.2	68.35
15	0.3	5.7	140.2	146.2	90,123.5	90,269.7	0.16

END

**DATE
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