Pilot-Scale Testing of the Hydrophobic-Hydrophilic Separation Process to Produce Value-Added Products from Waste Coal

NATIONAL ENERGY TECHNOLOGY LABORATORY

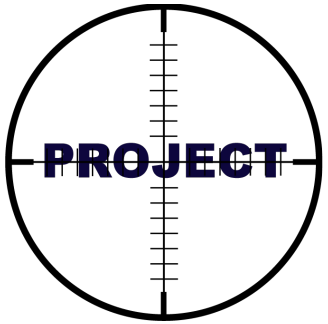
Jim Reyher
Minerals Refining Company
jreyher@mineralsrefining.com

Background

- In the cleaning of all minerals including coal, there is a limitation to the particle size that can be separated and cleaned mechanically. This limitation is approximately 325 mesh or 45 microns. Smaller particle sizes are either lost to a waste impoundment or recovered by filtration, which cannot provide marketable moisture levels. Another shortcoming with filtration is that it captures both the smaller mineral and the earthen matter particles.
- Minerals Refining Company (MRC) and Virginia Tech have jointly developed a hydrophobic-hydrophilic separation (HHS) process to cost effectively recover the less than 325 mesh coal particles.
- The HHS process has the capability to separate the coal particles from the earthen matter, resulting in an extremely low ash product.
- The HHS process also has the capability of recovering coal with single digit moistures using significantly less energy than conventional drying processes.
- MRC has demonstrated this process in the lab, in a small-scale demonstration process (50 pounds recovered product per hour) and in a larger scale demonstration process (750 pounds recovered product per hour).

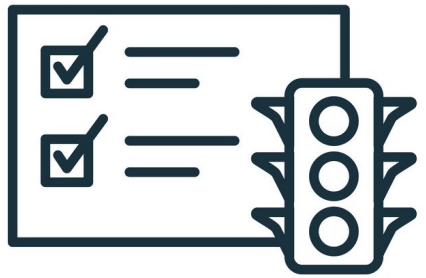
Project Scope

- In February 2019, the DOE approved a project to demonstrate the technical, economic and environmental benefits of the HHS process for producing high-purity, value-added clean coal and specialty carbon products from discarded coal waste.
- The project would use an existing eight-skid pilot-scale demonstration process to produce the following products:
  - Low ash (4-5%) and moisture (6-8%) coal from both bituminous and anthracite coal sources for use in conventional thermal and metallurgical coal applications.
  - Extremely low ash (<1.5%) and moisture (6-8%) coal from both bituminous and anthracite coal sources for use in high value-added specialty market applications. These low ash products would be produced in sufficient quantities for evaluation in potential customer applications.
- In addition, the project would evaluate the following process improvements to reduce the capital investment and operating costs associated with the process:
  - Reduce agglomeration energy
  - Increase particle size limit
  - Increase extraction column solids loading
  - Optimize solvent removal & recycle process
  - Evaluate coal briquetting
  - Minimize solvent losses



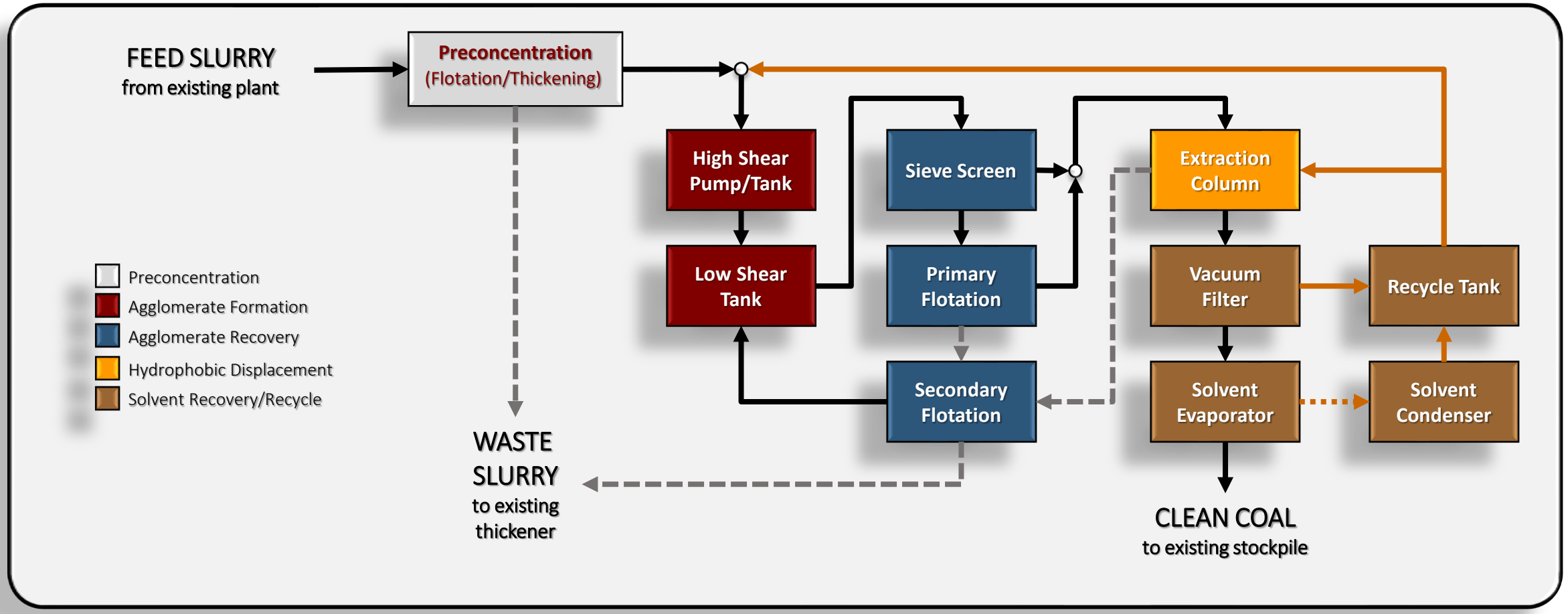
Project Status

- After project approval, the host facility for the eight-skid pilot-scale demonstration process decommitted from the project.
- The decommitment resulted in a revision to the project scope and the decision to use an existing one-skid pilot-scale demonstration process that had been developed for a DOE rare earth recovery project.
- In the third quarter of 2019, MRC signed an agreement to build the first commercial HHS coal recovery process at a metallurgical coal producing facility. Due to current capital and operating costs associated with the HHS process, projects can only be for higher value coal applications.
- Revised project scope was approved in May 2020.
- First commercial process will be mechanically complete in May 2021.

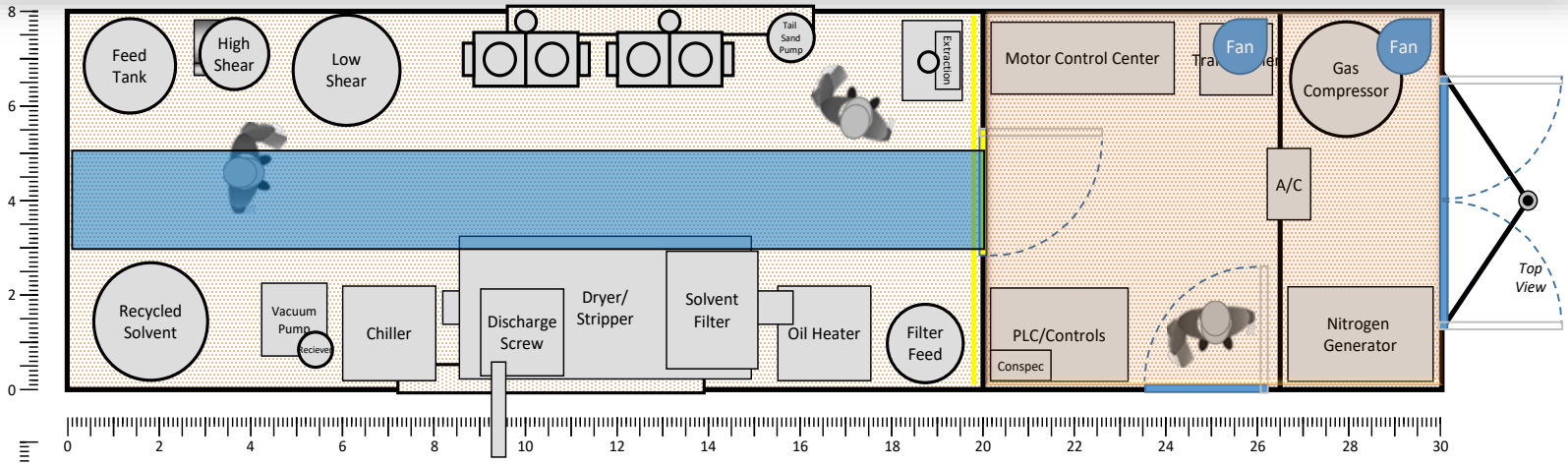


MRC Commercial & One-Skid Process

Commercial Process Flow Diagram



MRC One-Skid Demonstration Process

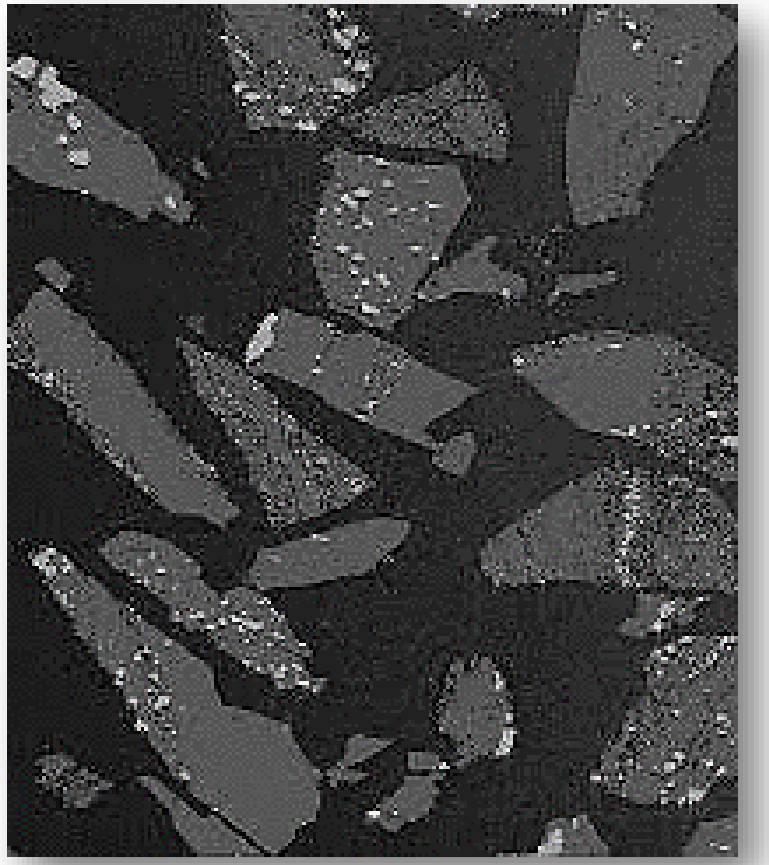


Project Status – Coal Recovery

- Due to the COVID-19 virus, a decision was made to process truckload-size quantities of coal slurry feedstock at Virginia Tech instead of taking the one-skid trailer to coal preparation facilities.
- Procedures were developed for handling the coal slurry feedstock and residual wastewater streams. These procedures were approved by Virginia Tech's Office of Environmental Health & Safety (EHS).
- Plan to process the following four feedstocks:
  - Bituminous waste coal slurry
  - Anthracite waste coal slurry
  - Coal recovered from impoundment pond
  - Finished coal product for low ash application
- Initial step is to evaluate the four feedstocks in the lab prior to processing through the one-skid process. Lab samples have arrived from two sources.

Project Status – Coal Recovery

- Production of low ash (<1.5%) coal will require grinding of the samples to adequately liberate mineral matter prior to processing through the one-skid HHS process. Previous lab testing has shown that not all coal slurry feedstocks will generate a lower ash coal product after grinding.
- Based on lab test results, extended trials will be conducted in the one-skid HHS process. Each trial will produce between 1,500 and 2,000 pounds of coal for application testing by potential customers.



Project Status – Process Improvement

- Reduce agglomeration energy:
  - Successfully demonstrated the capability to replace an expensive rotor/stator mixer with centrifugal pump for high shear energy input.
  - Currently working on optimizing high shear operating parameters (horsepower, pump rpm, impeller tip speed, pump circulating rate) with existing process.
  - Plan to evaluate two liquid flotation and micro agglomerate preconcentration as alternative agglomeration techniques.
- Increase particle size limit:
  - Existing process is limited to processing particle sizes less than 100 mesh through extraction column.
  - Currently evaluating replacement of the extraction column with vibrating jig for large particle size applications.
  - Vibrating jig successfully demonstrated in batch lab test. Next step is to scale-up to continuous lab test.
  - Based on success of lab testing, a pilot-scale jig will be tested in the one-skid HHS process.

Project Status – Process Improvement

- Increase extraction column solids loading:
  - Original commercial process design utilized a fixed bed structured packing extraction column. Due to the structured packing, there was always a risk for plugging of the extraction column with coal or mineral matter particles.
  - Recent testing has demonstrated that an extraction column with oscillating internals can be used to effectively replace the structured packing column.
  - Currently optimizing the operating parameters (oscillating frequency, agglomerate feed point, design of column internals, solvent velocity in the column, solids loading) associated with the oscillating column.
  - Maximizing the solids loading (coal concentration in solvent) will minimize the downstream solvent removal costs. Testing to date has demonstrated that solids loading can be increased from 7% to 9%.

Project Status – Process Improvement

Oscillating Extraction Column Internals

KARR® Column Plate Stack Assembly

Internals for a 3 foot (1 meter) diameter KARR® Column

KARR® internals installation

Koch Modular PROCESS SYSTEMS

Project Status – Process Improvement

- Optimize solvent removal & recycle process:
  - In the first commercial process, $2.8MM or 47% of the capital equipment associated with the HHS process is associated with the solvent removal and recycle process.
  - A separate lab project is currently evaluating the feasibility of using a pressure filter with steam stripping to eliminate the need for a solvent evaporator.
  - Based on success of this lab project, a pressure filter with steam stripping will be installed in the one-skid process to demonstrate the feasibility of this approach.
  - If successful, it will significantly reduce the capital investment for a commercial plant.
- Evaluate coal briquetting:
  - The coal recovered by the HHS process is typically 325 mesh or smaller. In some customer applications, this fine coal powder creates a dusting issue.
  - This project will investigate a cost-effective method to increase the size of the recovered coal particle.
  - No work has been initiated on this process improvement option.

Project Status – Process Improvement

- Minimize solvent losses:
  - The solvent used in the HHS process is isohexane.
  - From an environmental and cost perspective, it is critical to minimize the losses of solvent in the HHS process.
  - Environmental permitting requirements are based on amount of solvent losses.
  - The three main sources of solvent losses are:
    - Residual solvent in final product
    - Residual solvent in wastewater
    - Release of solvent from vent system and any fugitive (leak) emissions
  - Weekly samples are being taken from the one-skid process to measure solvent losses.
  - Process changes have been implemented to reduce solvent losses.

Summary

- Successful completion of this project will:
  - Identify new applications for use of the HHS process in the coal industry.
  - Reduce the capital investment and operating costs associated with the "second generation" HHS process. This will allow for the use of this technology in broader markets than the metallurgical coal market.
  - Provide an economic process for recovery of other minerals beyond coal.
  - Economically address environmental issues associated with impoundment ponds by providing a cost-effective method to recover and upgrade the waste coal.
  - Conserve natural resources by recovering materials that have already been mined.