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

NATIONAL ENERGY TECHNOLOGY LABORATORY

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U.S. DEPARTMENT OF ENERGY

MRC

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. The shortcomings with filtration are that it does not reduce the earthen matter content or the moisture content.
- 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

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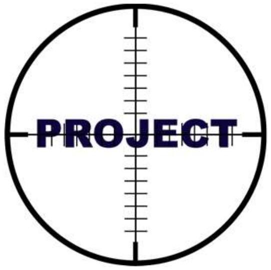
- In May 2020, the DOE approved a revised project to demonstrate the technical, economic and environmental benefits of the HHS process for producing high-purity, value-added clean coal and specialty carbon products from discarded coal waste.
- The project scope was revised to use an existing one-skid pilot-scale demonstration process to recover the following coal 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



Hydrophobic – Hydrophilic Separation Process

Step I – Collection

- Droplets of a low-boiling point solvent selectively coat hydrophobic particles.

Step II – Agglomeration

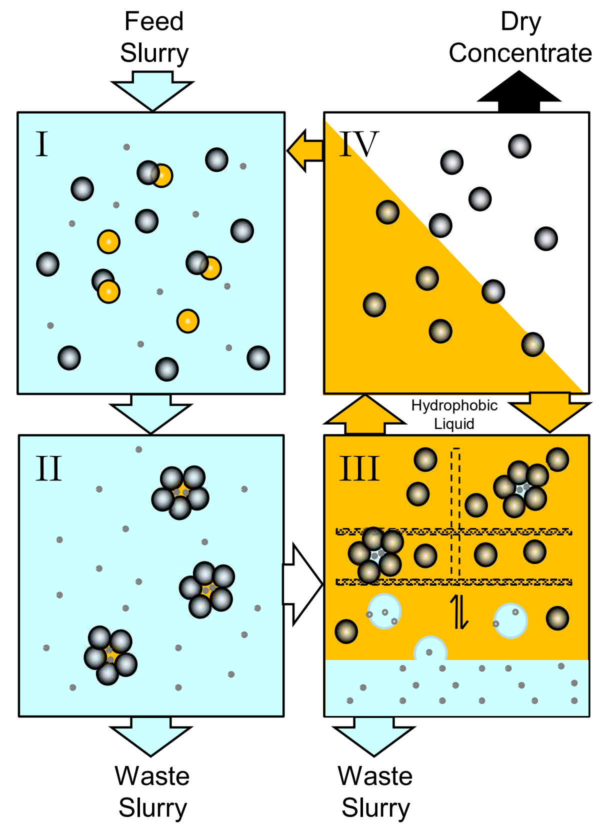
- The coated coal particles join together to form an agglomerate that can be separated from mineral matter.

Step III – Dispersion

- Agglomerates are broken in the presence of high concentration of solvent. Coal having a greater affinity for solvent than water remains in solvent phase. Water particles containing ash coalesce and separate from the solvent phase into an aqueous phase.

Step IV – Recovery/Recycle

- Spent solvent is recovered by filtration and evaporation for recycling in the process



MRC Commercial & One-Skid Process

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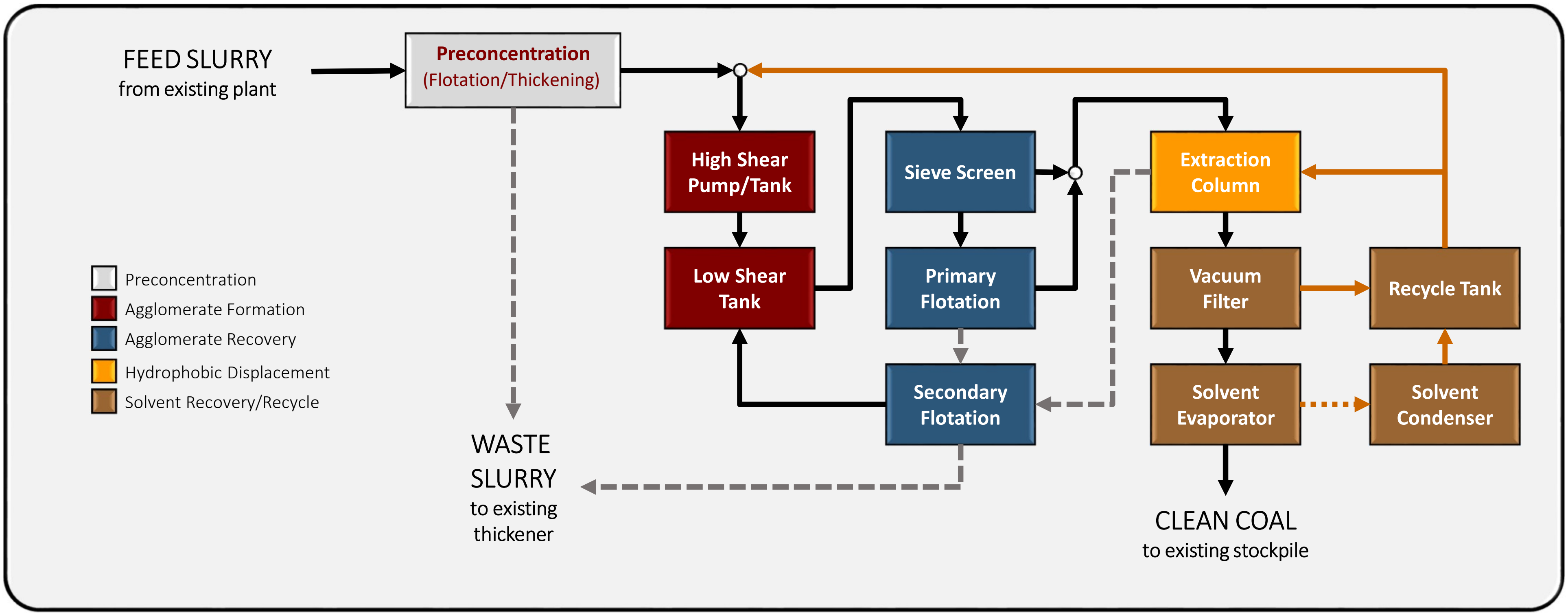
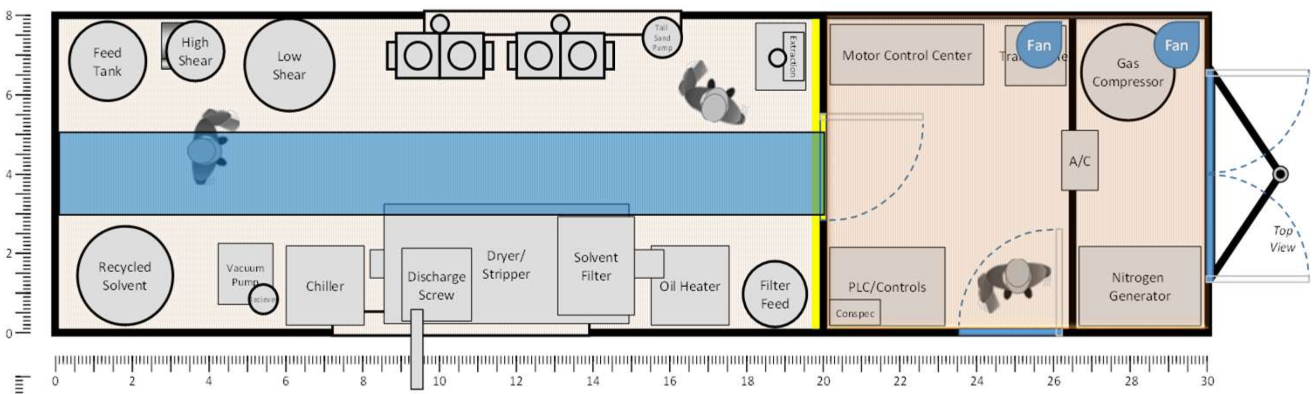
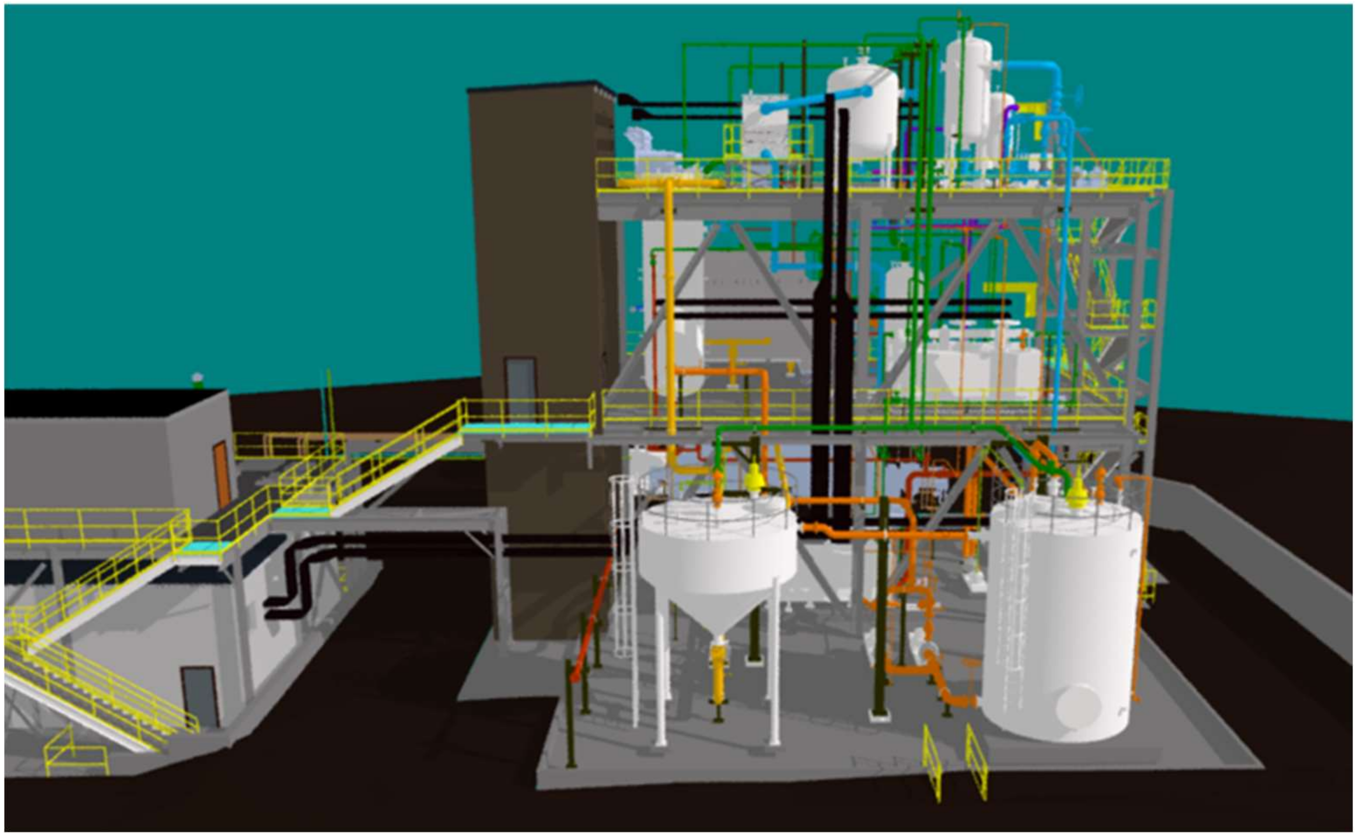
Commercial Process Flow Diagram



MRC One-Skid Demonstration Process



MRC Commercial Process – 3D Model



Project Status – Lab Evaluation

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Prior to recovering coal in the one-skid process, various coal slurry samples were evaluated in the lab HHS process. Results are as follows:

- Bituminous centrifuge effluent waste stream from a coal preparation facility with an ash concentration of 17% :
  - As received sample processed in lab HHS process resulted in coal with an ash concentration of 4.5% , moisture concentration below 8% and organic recovery of 98%.
  - Grinding of the coal to finer particle size did not result in a lower ash concentration.
- Bituminous waste impoundment coal sample after being processed through several grinding and flotation steps with ash concentration of 4.0% and D80 particle size of 14 micron:
  - As received sample processed in lab HHS process resulted in coal with an ash concentration of 1.8%, moisture concentration below 8% and organic recovery of 71%.
  - As received sample ground to 4 micron, processed through a flotation step and a lab HHS step resulting in coal with an ash concentration of 0.5 - 0.6%, moisture below 2% and organic recovery of 72 - 80%.

Project Status – Lab Evaluation

- Anthracite filtration feed waste stream from a coal preparation facility with an ash concentration of 61%:
  - As received sample processed in the lab HHS process did not result in good agglomerate formation. Investigation revealed that it was due to the presence of a high concentration of flocculant used to increase the coal slurry concentration prior to filtration.

- Anthracite waste impoundment sample with an ash concentration of 58%:
  - As received sample processed in lab HHS process did not result in good agglomeration formation.
  - As received sample was ground to 25 micron, processed through flotation step and lab HHS step resulting in coal with an ash concentration of 3.0%, moisture 9.5% and overall organic recovery of 51%.

Project Status – One Skid HHS Trials

- Numerous one-skid HHS trials have successfully been completed using the bituminous centrifuge effluent waste stream from a coal preparation facility. The recovered coal had an ash concentration of 4.5% which was equivalent to the lab HHS ash results.

- The bituminous centrifuge effluent waste stream has been used for all process improvement testing to ensure a consistent source of feed.

- When lab HHS evaluations are complete, one-skid HHS trials will be completed using the following waste coal slurry streams:
  - Bituminous waste coal slurry from an impoundment to produce less than 1.5% ash product.
  - Anthracite waste coal slurry stream from an impoundment to produce both a less than 4.5% ash product and a less than 1.5% ash product.

Project Status – Process Improvement

- Reduce agglomeration energy:
  - Extensive one-skid HHS testing has determined the high and low shear energy requirements for processing an 8% solids coal slurry feed:
    - High shear total shaft energy: 0.22 HP per gpm.
    - High shear hydraulic energy: 0.075 HP per gpm.
    - High shear tip speed: 44.3 feet per second minimum
    - High shear circulating rate: 5 times the coal slurry feed rate.
    - Low shear energy: 0.015 HP per gpm.
  - Lab HHS testing has shown that the high shear energy requirements increase with increasing coal slurry feed concentration. This increase needs to be quantified with one-skid HHS process testing.

Project Status – Process Improvement

- Reduce agglomeration energy:
  - Lab HHS testing has shown that flocculant addition used to increase the coal slurry feed solids concentration has a negative impact on agglomerate formation. This impact was minimized in the lab by increasing the high shear agglomeration energy and/or addition of a hydrophobicity enhancing agent. One-skid HHS process testing will need to be completed to verify that the negative impact can be mitigated.
  - One-skid HHS testing successfully demonstrated the capability to replace the Silverson high shear mixing pumps with high shear centrifugal pumps which will result in $175,000 reduction in capital investment for commercial process.
  - Plan to evaluate two-liquid flotation and micro agglomerate preconcentration as alternative agglomeration techniques.

Project Status – Process Improvement

- Increase particle size limit:
  - Existing process is limited to processing particle sizes less than 100 mesh due to limitations on the upward velocity in the 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.
  - Based on success of lab testing, a pilot-scale jig will be tested in the one-skid HHS process.

Project Status – Process Improvement

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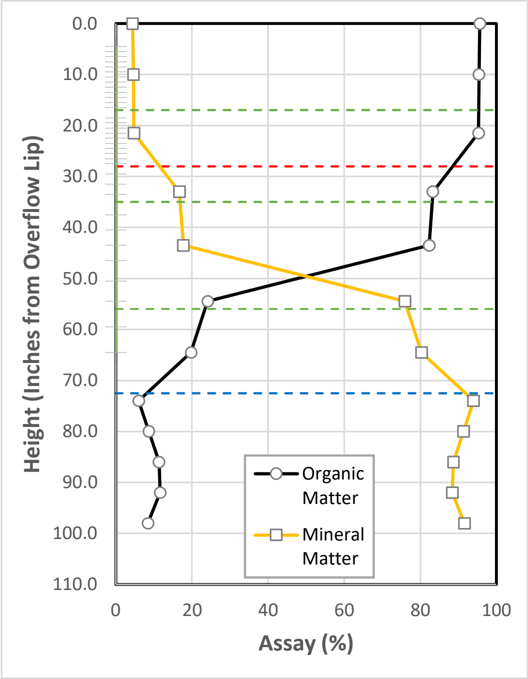
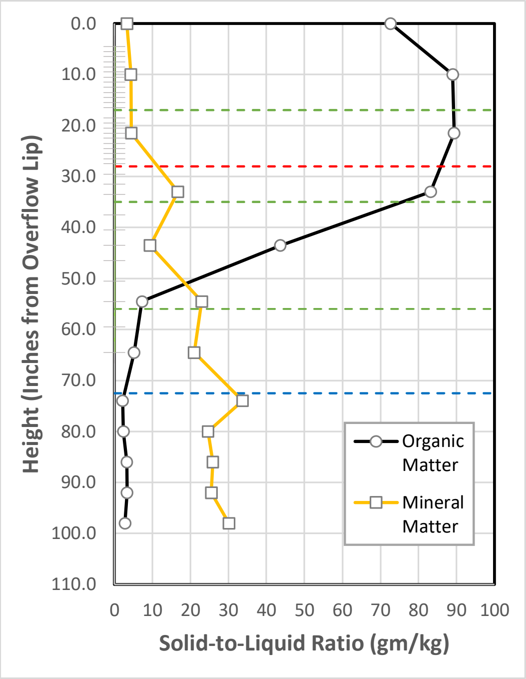
- Increase extraction column solids loading:
  - Original commercial process design utilized a fixed bed structured packing extraction column. Due to the structured packing, there was always a risk for plugging of the extraction column with coal or mineral matter particles.
  - Testing has demonstrated that an extraction column with oscillating internals can be used to effectively replace the structured packing column.
  - Based on extensive testing in the one skid process, the design parameters (oscillating frequency, agglomerate feed point, design of column internals, solvent velocity in the column, solids loading) associated with the oscillating column have been optimized.
  - 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

- Increase extraction column solids loading (column profiles):





Project Status – Process Improvement

- Optimize solvent removal & recycle process:
  - In the first commercial process, $2.5MM or 41% of the capital equipment associated with the HHS process is used in the solvent filtration and evaporation steps.
  - A separate DOE project has demonstrated that either a pressure filter or a vacuum filter with steam stripping can effectively remove the solvent from the coal using only the filtration step. Residual solvent concentrations less than 500ppm were achieved with either pressure or vacuum filtration with steam stripping.
  - Based on success of this lab project, a pressure filter or vacuum filter with steam stripping will be installed in the one-skid process to demonstrate the commercial viability of this approach.
  - If successful, it will significantly reduce the capital investment for a commercial HHS process.

Project Status – Process Improvement

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- 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

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- Minimize solvent losses
  - From an environmental and cost perspective, it is critical to minimize the losses of solvent in the HHS process.
  - 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
  - For the final product, the residual solvent concentration averaged 250ppm with range of 10 to 680 ppm. The recycle gas system associated with the solvent evaporator will be optimized to further reduce this source of solvent losses.
  - For the wastewater, the residual solvent concentration averaged 28ppm with range of 6 to 88 ppm. The secondary solvent recovery flotation unit will be optimized to further reduce this source of 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.

- Thank you to the Department of Energy for their support and assistance with this project.

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