

# **Pilot-Scale Testing of the Hydrophobic-Hydrophilic Separation (HHS) Process to Produce Value-Added Products from Waste Coals (FE00317111)**

by

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*October 27, 2022  
2:15-2:45 pm*

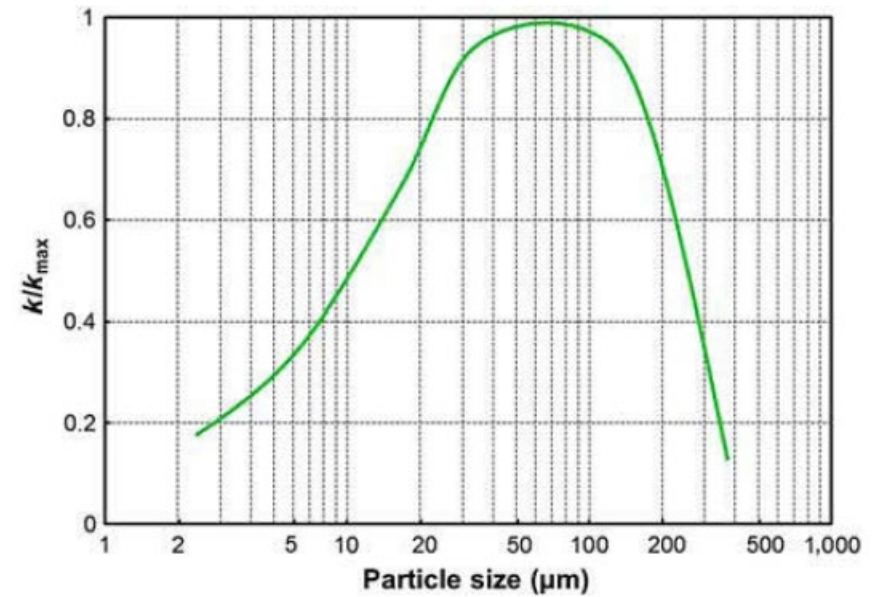
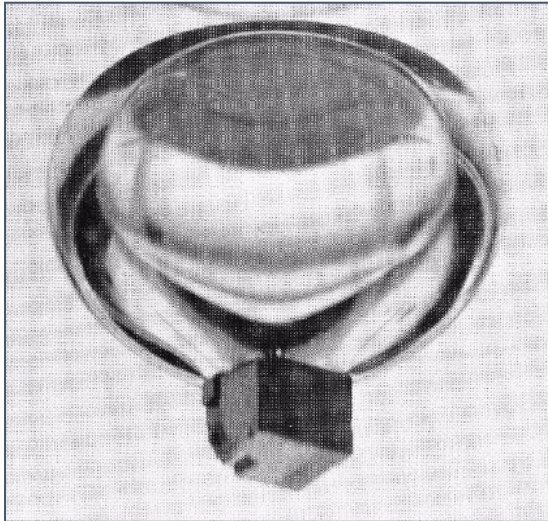
# Project Overview

- ❑ Funding
  - DOE \$1,445,486
  - Cost Share \$361,371
- ❑ Objective
  - To produce low-ash feedstocks for high-value carbon products
- ❑ Prime
  - Minerals Refining Company (MRC, Inc.), Richmond, VA
  - Dr. Stan Suboleski, PI
- ❑ Sub
  - Virginia Tech
    - *Roe-Hoan Yoon, Co-PI*
    - *Aaron Noble, Co-PI*
- ❑ Project Manager
  - Christian Robinson
  - End date
    - *December 31, 2022*

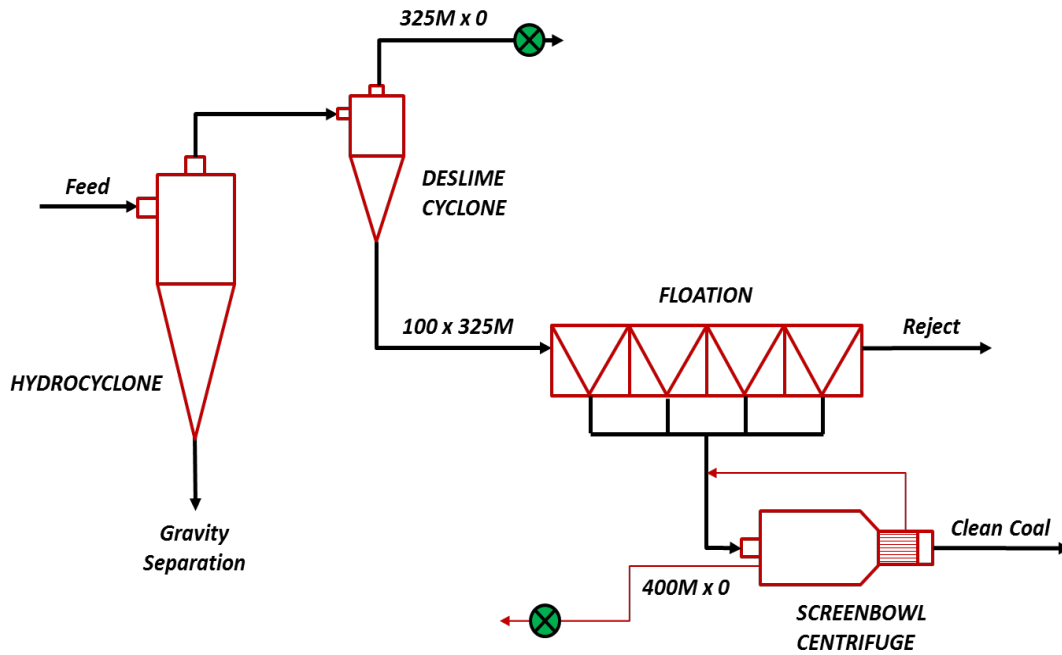
# Flotation

## □ Produces all metals humans use

- Sulman and Piccard (1905)
- US Patent 793,808

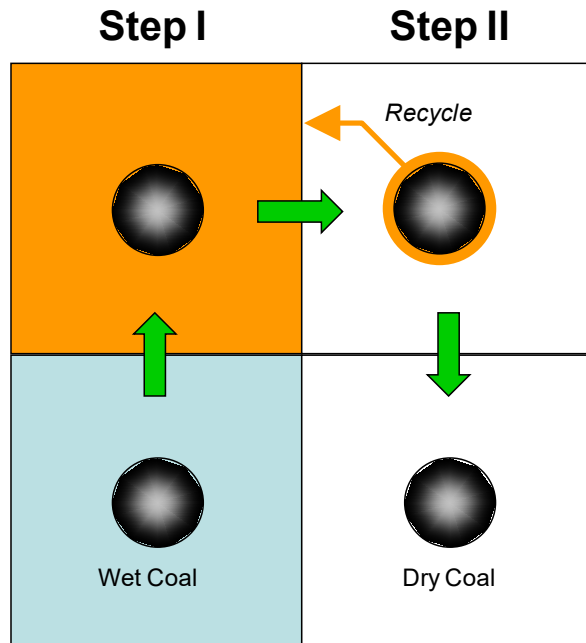


# Fine Coal Cleaning in the US



# Dewatering by Displacement (DbD)

Yoon and Luttrell, US Patent 587,085 (1995)

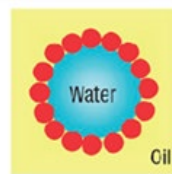
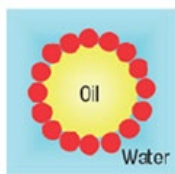
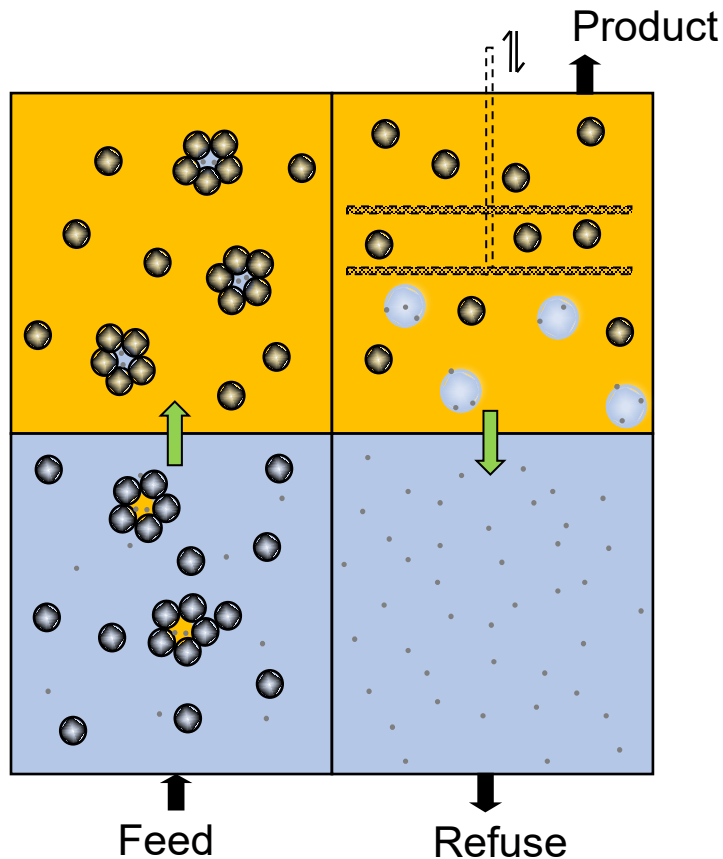


Liquid	Boiling Point <sup>1</sup> (°C)	Heat of Vaporization	
		kJ/mole <sup>2</sup>	kJ/kg
Water (H <sub>2</sub> O)	100.0	40.7	2,257
Butane (C <sub>4</sub> H <sub>10</sub> )	-0.5	22.4	386
Pentane (C <sub>5</sub> H <sub>12</sub> )	36.1	25.8	358
Hexane (C <sub>6</sub> H <sub>14</sub> )	68.7	28.9	336
Heptane (C <sub>7</sub> H <sub>16</sub> )	98.4	31.7	317

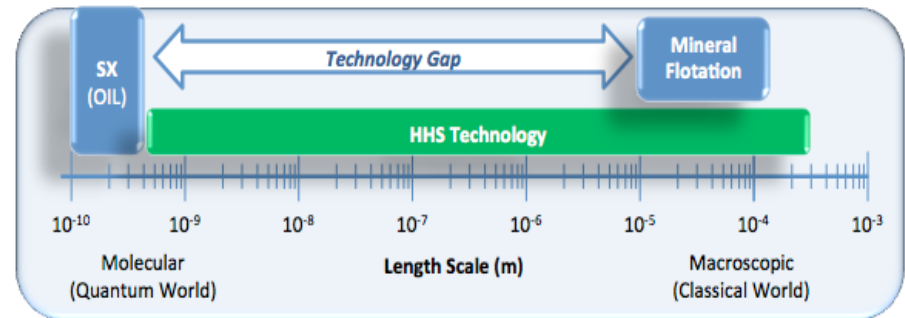
$$\Delta G_t = \gamma_{wo} \cos \theta$$
$$< 0$$

# Hydrophobic-Hydrophilic Separation (DHS)

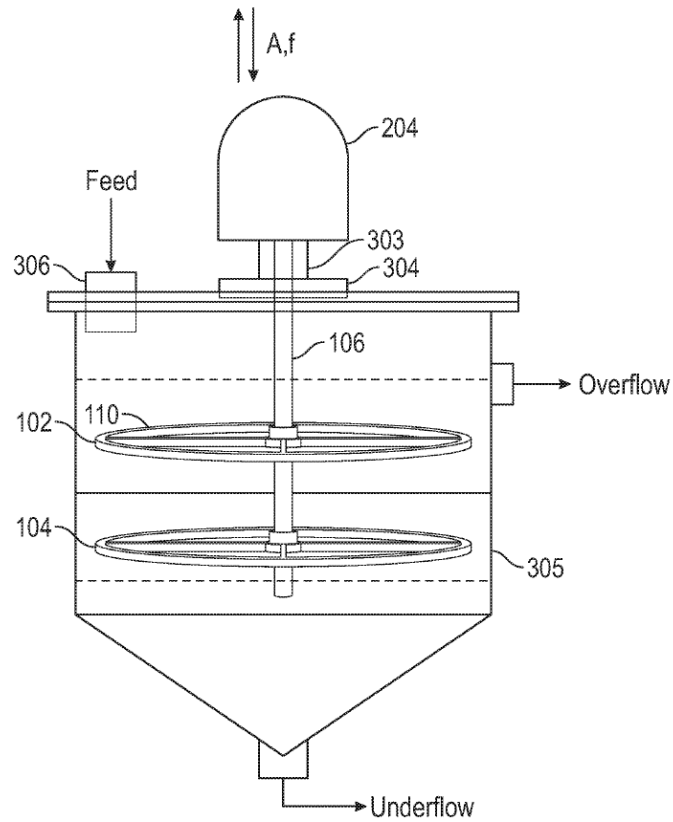
Yoon, 2016; Yoon *et al.* (2017)



- ❑ No lower particle size limit
  - High contact angle
- ❑ High grade
  - MORGANIZER
- ❑ Dry product
  - DbD



# Screen Morganizer





# HHS POC Unit at VT





# POC-Scale Test Results

Coal Samples & Location	Feed Ash (%wt.)	HHS Product (%wt.)		Reject Ash (%wt.)	Organic Recovery (%wt.)	Sample Type
		Moisture	Ash			
Eastern Kentucky Mine A – PCI/Thermal	67.5	6.8	3.8	88.3	91.6	Deslime Cyclone OF
Southern WV Mine B – High Vol Met	53.0	8.5	3.4	84.4	89.5	Deslime Cyclone OF
Western Pennsylvania Mine C – Thermal Pitt 8	40.4	3.9	4.3	85.8	95.5	Screen Bowl Effluent
Southern WV Mine D – High Vol Met	53.6	3.5	3.9	88.0	95.4	Deslime Cyclone OF
Northern WV Mine E – High Vol Met	13.7	4.9	5.0	76.3	97.7	Screen Bowl Effluent
SW Virginia Mine F – Low Vol Met	7.0	3.8	2.1	64.8	97.6	Screen Bowl Effluent
Southern WV Mine G – Low Vol Met	10.7	3.8	3.1	79.4	98.6	Screen Bowl Effluent

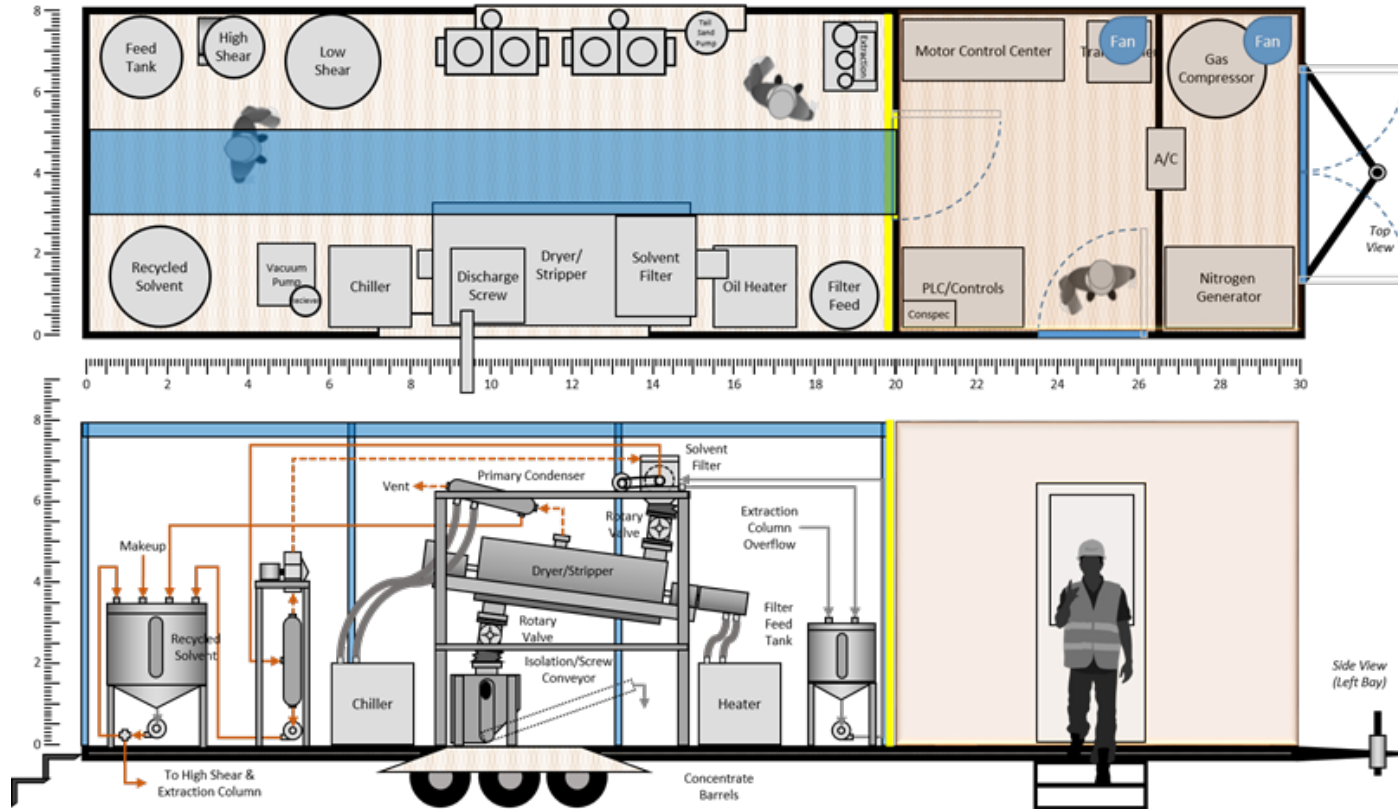
# HHS Pilot Plant

(1 tph in Virginia)



No binder

# One-Skid Pilot Plant



Initial design concept of One-Skid HHS Pilot Plant



# One-Skid Pilot Plant



*Front*



*Back*

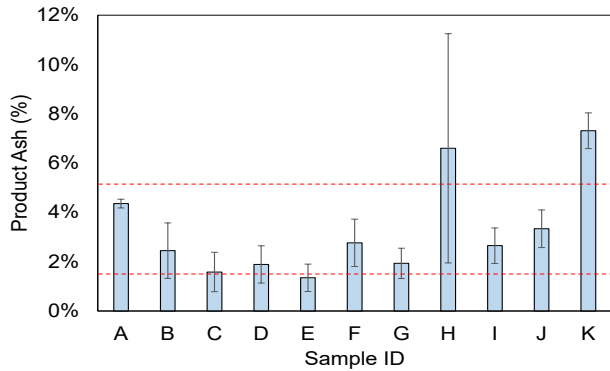
# Lab Tests for Warrior Met Coal

Sample Type	Feed Ash (%wt.)	HHS Product (%wt.)		Reject Ash (%wt.)	Organic Recovery (%wt.)
		Moisture	Ash		
Screen Bowl Effluent	18.9	1.2	4.1	89.5	99.3
Screen Bowl Effluent	16.2	1.6	3.9	87.6	99.0
Thickener Under Flow <sup>1</sup>	49.7	8.4	5.3	82.5	86.5
Thickener Under Flow	54.9	4.8	6.6	85.8	88.9
Pond Sample	50.4	4.3	7.6	88.8	95.3

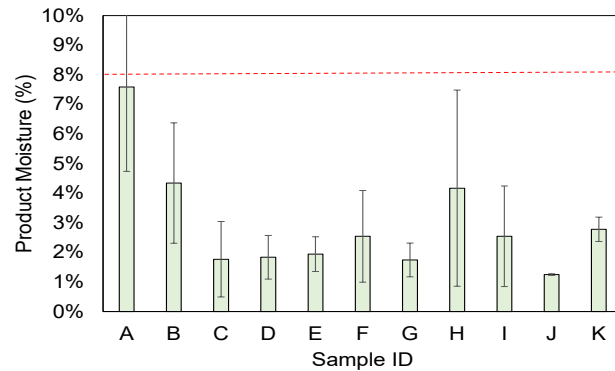
<sup>1</sup>Pre-screened at 100 mesh (150  $\mu$ m)



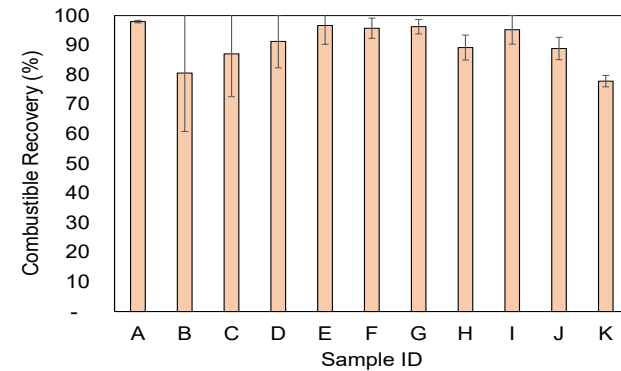
# Lab Test Results



*Product Ash.*  
Dashed lines show upper threshold limits for super-clean (5%) and ultra-clean (1.5%) products.



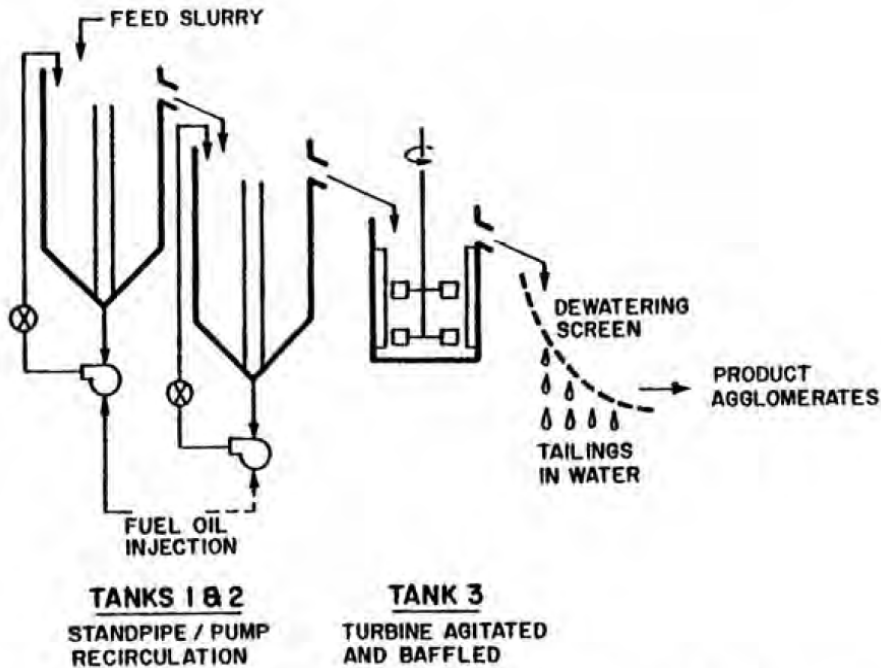
*Product Moisture.*  
Dashed lines show upper threshold limits for moisture objectives



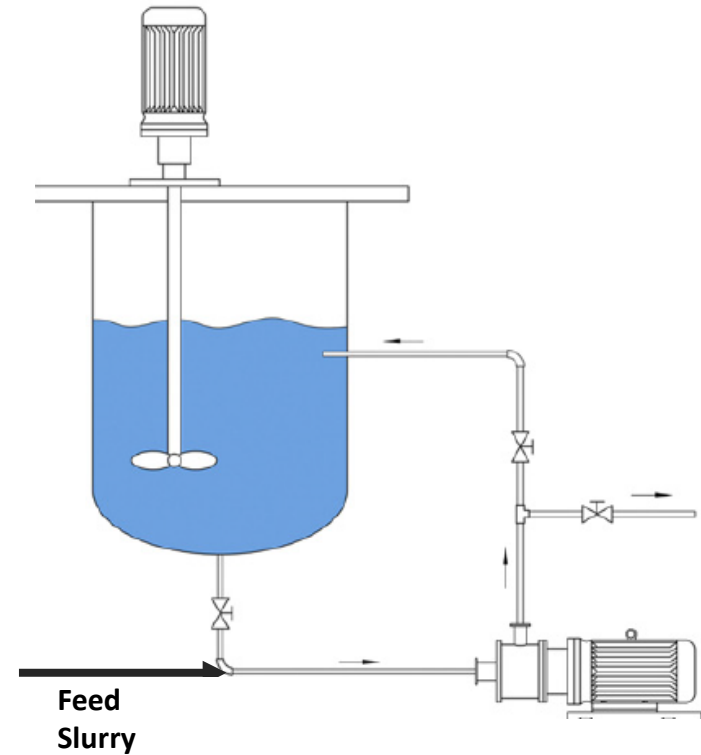
*Combustible Recovery.*

# Mixing System

- Standpipe + pump
  - Capes (1979)



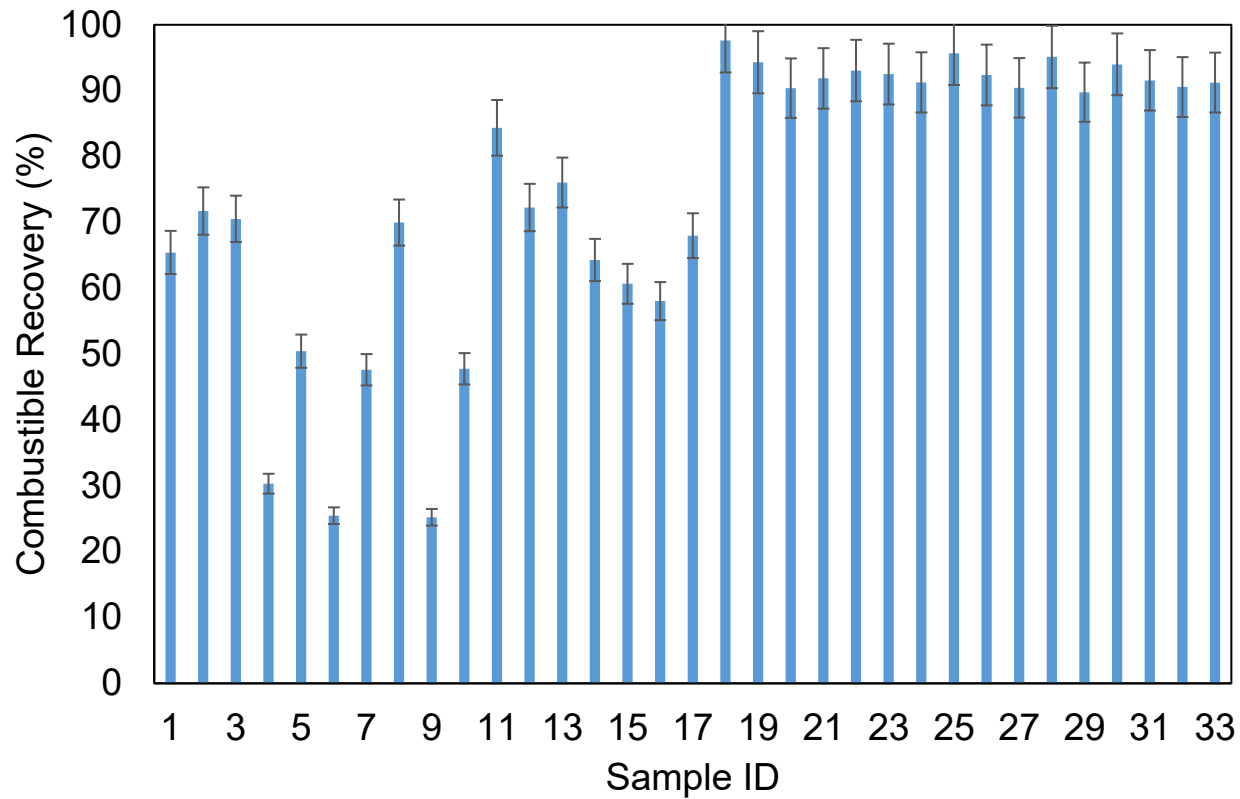
- In-line HSM + impeller
  - Zhang *et al.* (2012)



$$R = \frac{k\tau}{1 + k\tau}$$

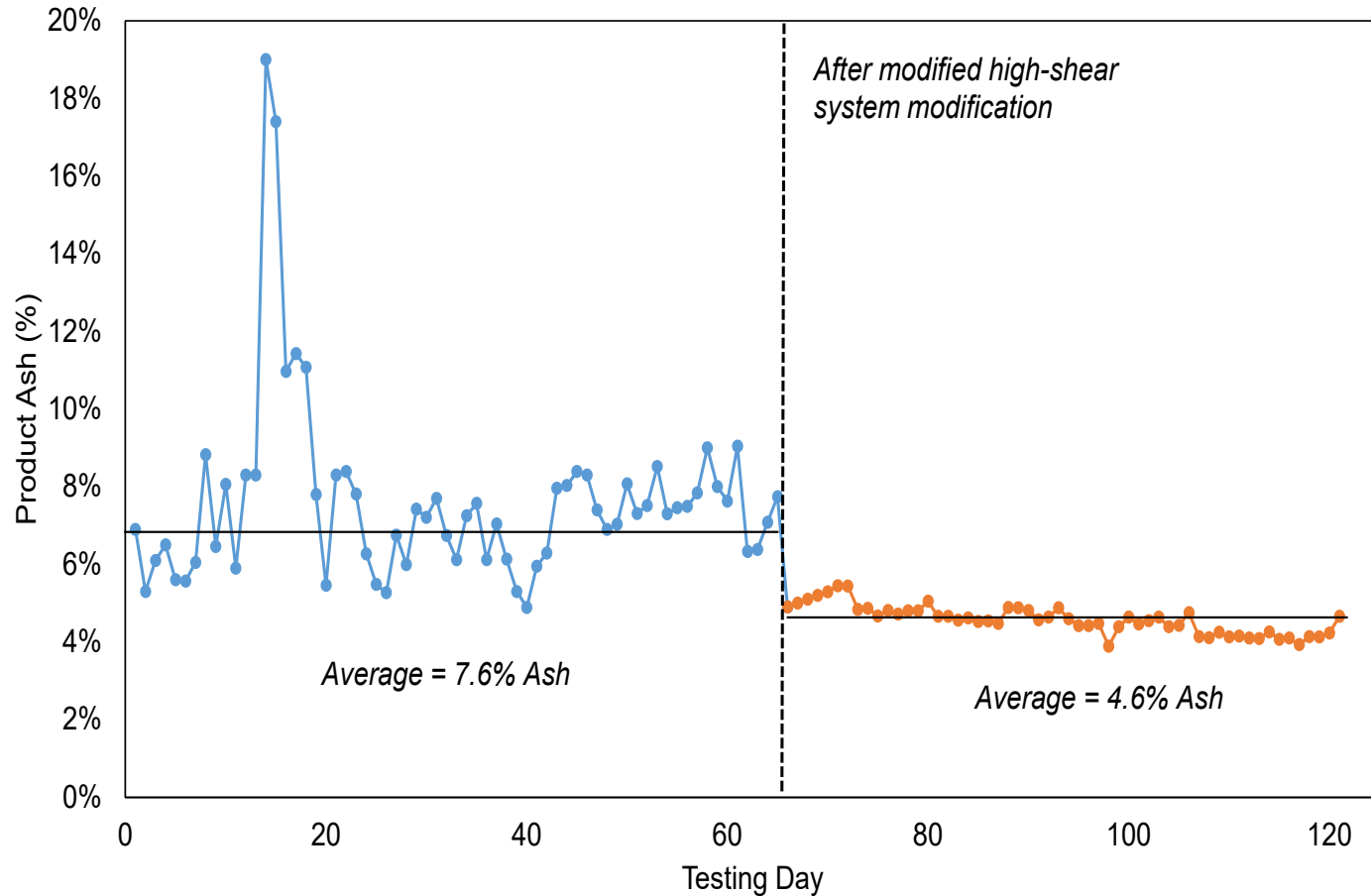
# Pilot Plant HHS Testing (Recovery)

- Before and after retrofit of agglomeration unit



# Pilot Plant HHS Testing (Product Ash)

- Before and after retrofit of agglomeration unit



# First Commercial Plant





# Vibrating-Screen Morganizer



# Night View Worrier Met, Alabama



# Testing Results: Ultraclean – Bituminous

- ❑ Tested a decanter effluent sample from a West Virginia bituminous processing plant.
- ❑ Product ash content of <1.5% was obtained on a sample from December trials.
- ❑ Work is continuing to create additional ultraclean products for end-user product testing.

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<b>Pilot Test ID</b>	<b>Product Ash</b>	<b>Product Moisture</b>
S-1	2.26%	3.25%
S-2	2.20%	20.1%
S-3	1.66%	2.70%
S-4	1.46%	2.80%
S-5	1.64%	12.7%

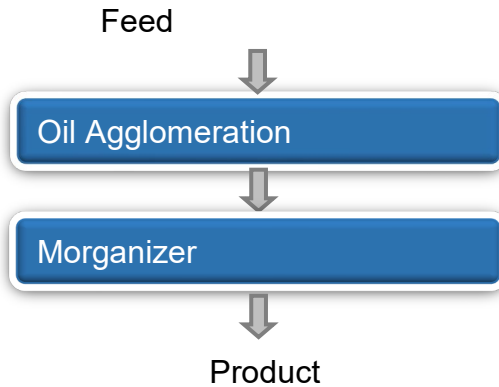
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# HHS vs. Two-Liquid Flotation (TLF)

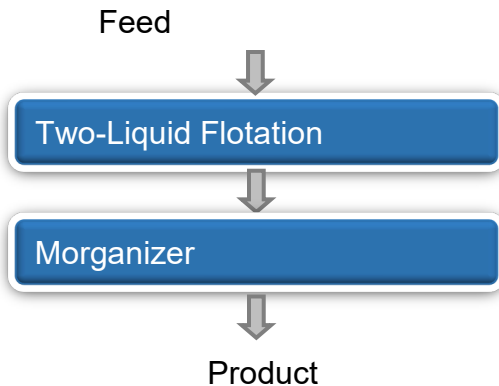
## (Results obtained on Clean Anthracite Sample)

### Process flow diagram

#### HHS



#### TLF



### Advantages of TLF

- Low energy consumption
  - *High-shear mixing is not required*
- Morganizer is more efficient
  - *Low ash content in the product*
- Simple flowsheet

Separation Method	Feed $D_{80}$ ( $\mu\text{m}$ )	Feed Ash (%)	Product		
			Ash (%) <sup>1</sup>	Moisture (%)	Organic Recovery (%)
HHS	11 <sup>1</sup>	11.53	2.19	1.83	92.53
TLF			1.49	2.68	79.73
HHS	10 <sup>2</sup>	6.34	2.21	2.32	97.48
TLF			1.46	2.93	93.56

<sup>1</sup>The as-received sample was ground.

<sup>2</sup>The as-received sample was ground and pre-concentrated.



# Ultraclean Coal

## (Arq)

### Flotation product

- The 2<sup>nd</sup>-stage flotation product

HHS Feed D <sub>80</sub> * (µm)	HHS Feed Ash (%)	Proprietary Reagents	HHS Product		Refuse Ash (%)	HHS Organic Recovery (%)
			Ash** (%)	Moisture (%)		
4.9	5.69	Reagent B	0.91 <sup>4</sup>	1.50	79.26	99.06
4.9	5.69	Reagent C	0.72 <sup>4</sup>	1.40	81.31	99.20
4.9	5.69	Reagent D	0.66 <sup>4</sup>	1.50	81.13	99.18

CCN2316

### Agglomeration product

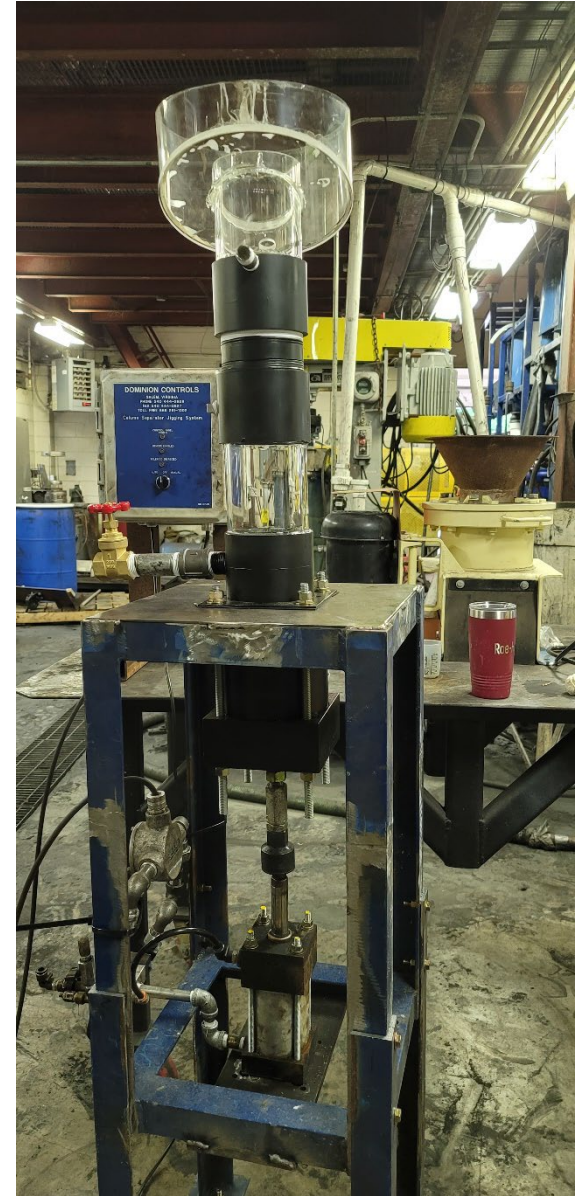
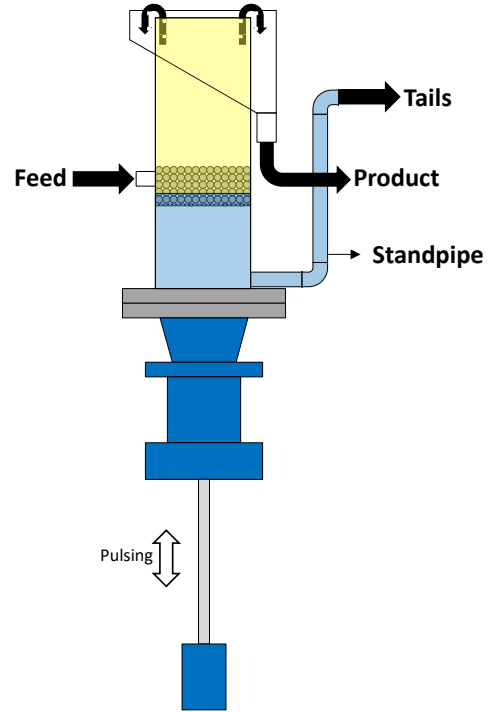
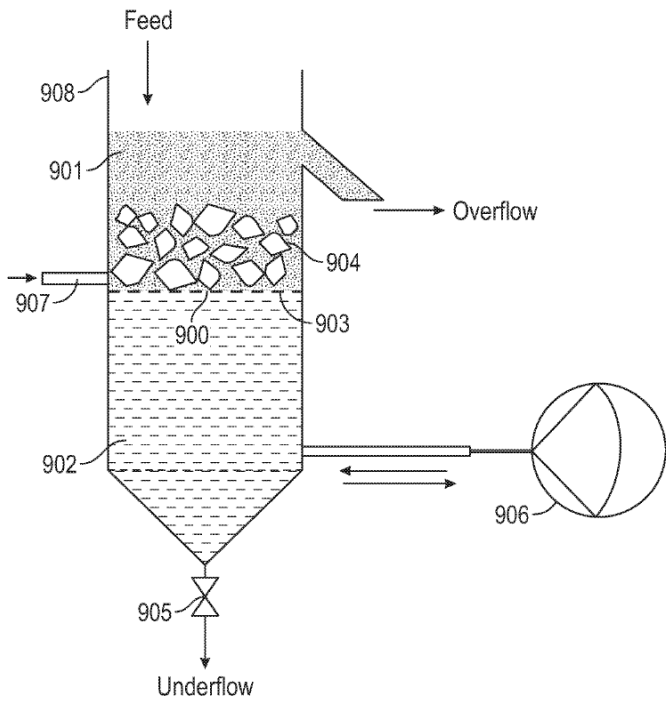
- Single-stage agglomeration

HHS Feed		HHS Product		Organic Recovery (%)	
D <sub>80</sub> (µm)	Ash (%)	Ash (%)	Moisture (%)	HHS	Overall
3.6	5.89	0.80	1.98	95.23	88.00

CCN5357



# Jig Morganizer

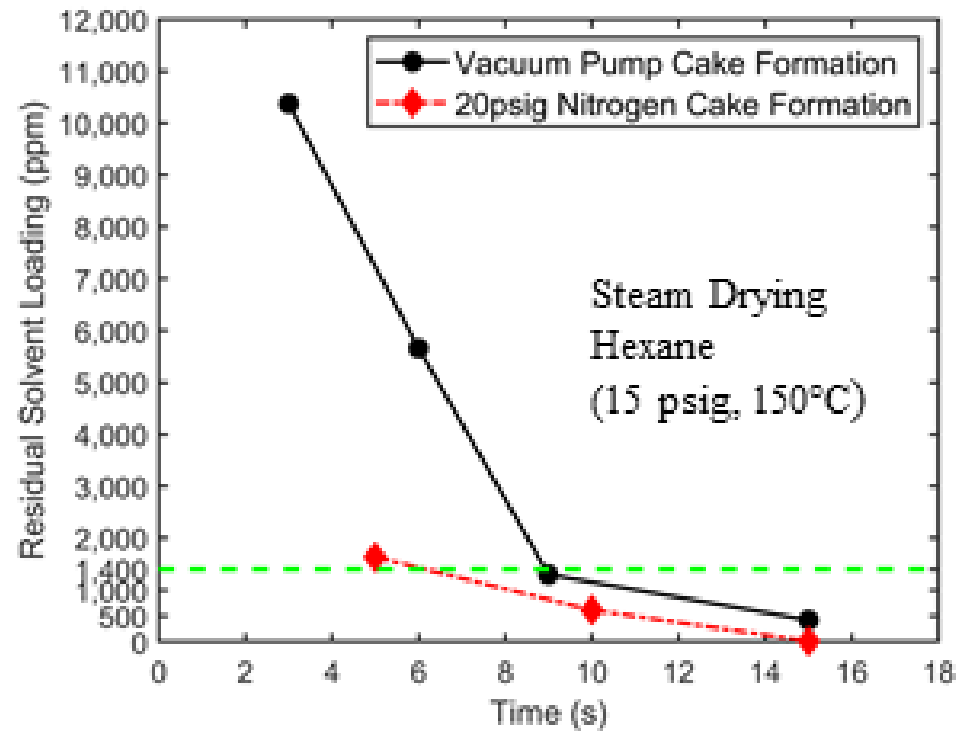


# Solvent Recovery

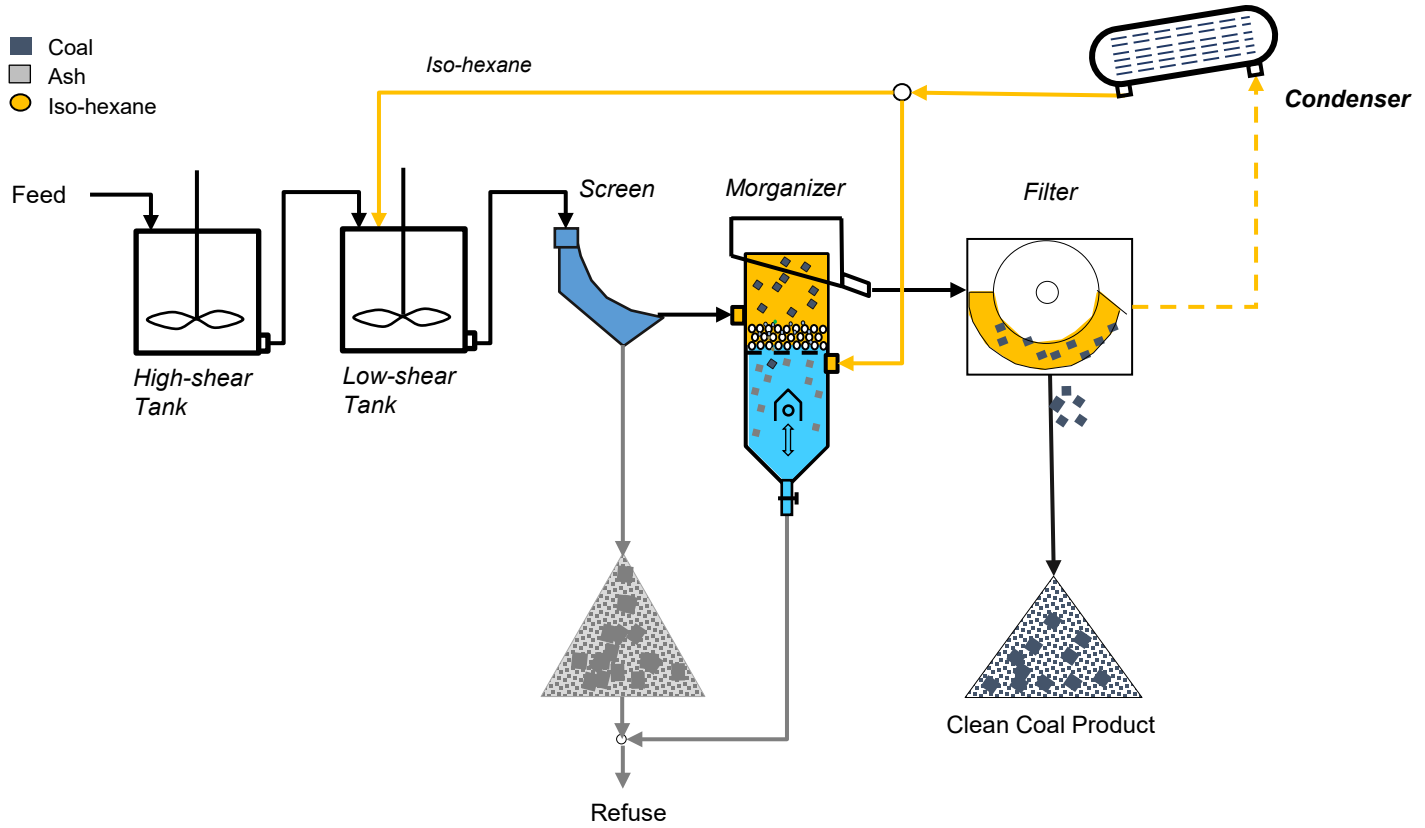
## (by Steam Stripping)



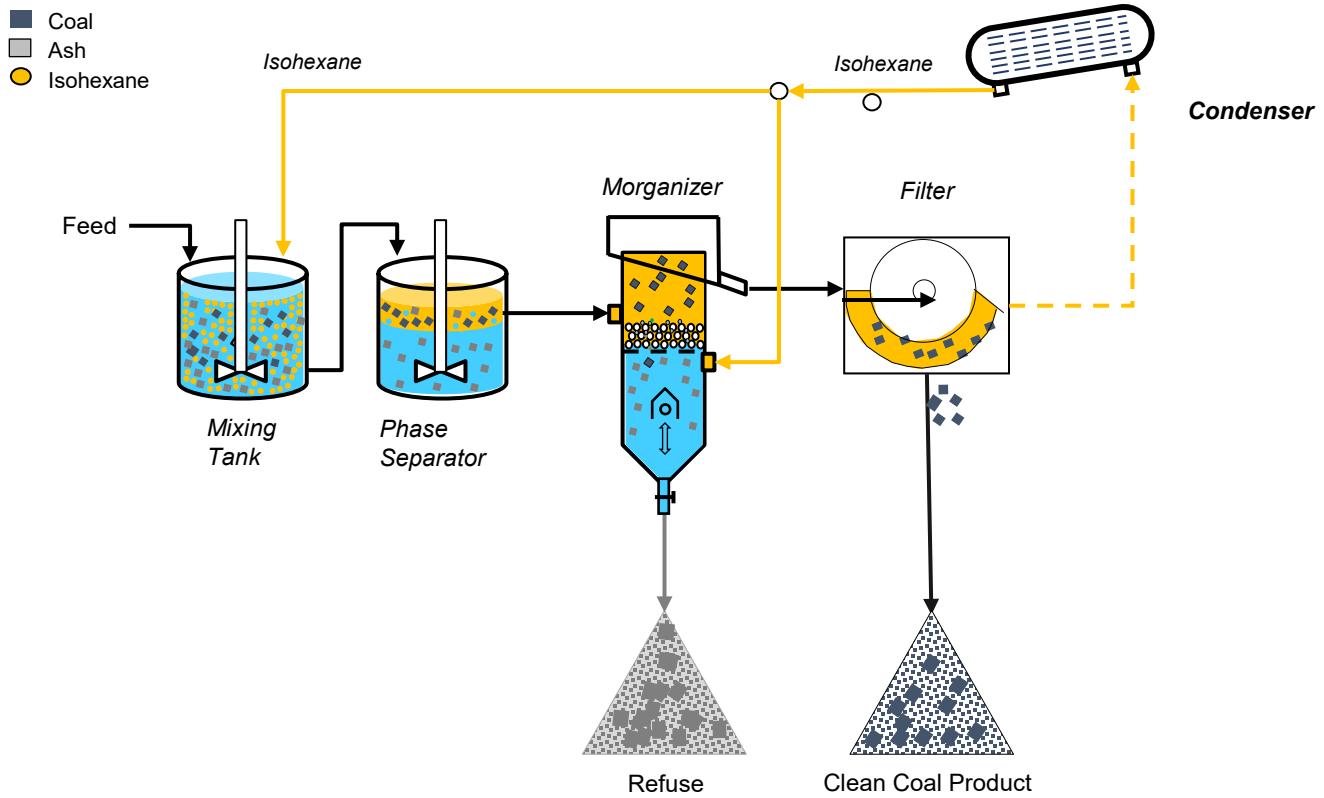
### □ Vacuum Filter



# HHS Flowsheet



# TLF<sup>1</sup> Flowsheet



<sup>1</sup>Two-liquid flotation

# Summary

- ❑ Virginia Tech has developed the HHS process
  - Remove mineral matter and surface moisture simultaneously
  - No lower particle size limit
  - Produces practically dry products
- ❑ Mobile pilot-scale test unit has been designed and constructed.
  - Tested successfully to produce salable coal from fine coal wastes
    - <5% ash, <7% moisture
  - Can produce low-ash and low-moisture feedstocks to produce high-value carbon products
    - <1.0 to 1.5% ash
- ❑ The pilot-scale test unit has been used to collect the scale-up information
  - First commercial plant near completion.
- ❑ Further improvements have been made
  - Jig Organizer
  - Steam stripper
  - two-liquid flotation (TLF) process
- ❑ Outcome
  - Lower capital & operating costs
  - Modular unit development
  - Broader commercial deployment