

AN OVERVIEW OF SOLID–LIQUID SEPARATION OF RESIDUES FROM COAL LIQUEFACTION PROCESSES

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Direct coal liquefaction process typically produces mixed oils (60%) and gases (15%). The remainder is a high-boiling viscous residue that contains oils, asphaltenes, unreacted coal, mineral matter and potentially valuable liquefaction catalyst. Effective separation of the components of the residue stream is important to the economic and environmental performance of the process. Solid–liquid separation technologies, such as filtration, hydrocyclones, centrifugation, critical solvent deashing and distillation have been reviewed in relation to their use in coal liquefaction processes. Individual operations used have not been completely satisfactory, and a better overall result is obtained when they are used in combination.

Keywords: coal liquefaction residue, solid–liquid separation, hydrocyclones

INTRODUCTION

Direct coal liquefaction (DCL) is a process for converting coal to liquid fuels. It involves dissolving coal in an organic (hydrogen donor) solvent in the presence of hydrogen gas in a reactor under moderate temperatures (400–450°C) and high pressures (200–300 bar). The liquefaction product can be broadly separated into gases (~15%), mixed oils (~60%) and residue (~25%). The dominant product is the mixed oil fraction, which after further processing in refinery units, produces gasoline, aviation turbine and diesel fuels. The product gases can be processed to recover hydrogen, which is recycled back to the reactor system. The utilisation of residue, which is usually the bottoms stream from separator stage or a vacuum distillation step, is ultimately critical to the economic and environmental impact of coal liquefaction processes.

In coal liquefaction processes, the residue, such as petroleum residue, is typically dark in appearance and highly viscous. However, unlike petroleum residue, DCL residues are more chemically complex and their processing and use is much less well developed.

Coal liquefaction technologies have been developed with different process objectives, giving different types of products. Some technologies have been developed to produce a clean fuel for power generation, as in the case of the solvent refined coal process stage-1 (SRC I), with the coal-derived liquid being a by-product. Other processes were aimed at maximising distillable liquid products, which are used for transportation fuels.

Several direct liquefaction technologies with different process configurations are described in the literature (Kamal, 1999).

Coal–liquid yields have improved as these technologies have progressed from single-stage to two-stage liquefaction processes. The main technologies developed as single- and two-stage processes are shown in Table 1.

Commercial-scale production of synthetic fuel by DCL (Shenhua SH-I first stage 24,000 bbl/day) is presently underway in Inner Mongolia, China (ASME, 2010; files.asme.org/asmeorg/Communities/Technical/Energy/16089.pdf, accessed 2nd Oct. 2011). The Shenhua process is based on the Southern Company Inc. catalytic two-stage liquefaction (CTSL) continuous flow process (Comolli et al., 1999). Figure 1 shows a schematic of the Shenhua coal liquefaction process.

Abbreviations: BCC, British Coal Corporation; BCL, brown coal liquefaction; CC-ITSL, close-coupled integrated two-stage liquefaction process; CCLP, Chevron coal liquefaction process; COED, char oil energy development; CSF, consol synthetic fuel; CTSL, catalytic two-stage liquefaction process; EDS, Exxon donor solvent; FMC Corp, Food Machinery and Chemical Corporation; GDC, Gas Developments Corporation; H-Coal, hyper-coal; ITSL, integrated two-stage liquefaction; LSE, liquid solvent extraction; NEDO/L, New Energy and Industrial Technology Development Organisation/Limited.

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Table 1. Coal liquefaction technologies*

Direct coal liquefaction processes and developer*	Number of stages	Years of activity	Operational size (TPD, coal)
SRC-II (Gulf Oil, USA)	Single	1965–1992	50
Conoco zinc chloride (Conoco, USA)	Single	1970–1980	1
NEDOL (NEDO, Japan)	Single	1978–1987	150 (Commercial ready)
H-Coal (HRI, USA)	Single	1980–1983	200
EDS (Exxon, USA)	Single	1980–1985	250
Kohleol (Ruhrkohle, Germany)	Single	1981–1987	200
Imhausen (Germany)	Single	1982–1984	0.1
CSF (Consolidation Coal Co., USA)	Two	1963–1970	70
CCLP (Chevron, USA)	Two	1970–1983	5
Dow Coal Liquefaction (USA)	Two	1970–1980	0.1
Lummus ITSL (Lummus Crest, USA)	Two	1972–1982	0.3
LSE (BCC, UK)	Two	1973–1995	2.5
CTSL/CC-ITSL (Southern Company Inc., USA)	Two	1982–1992	3–6 (Commercial ready)
BCL (NEDO, Japan)	Two	1985–1990	50 (Commercial ready)
Shenhua SH-I (Shenhua Group, China)	Two	2008–ongoing	3000 (Commercial—first operational plant)

* Source: see Abbreviations list.

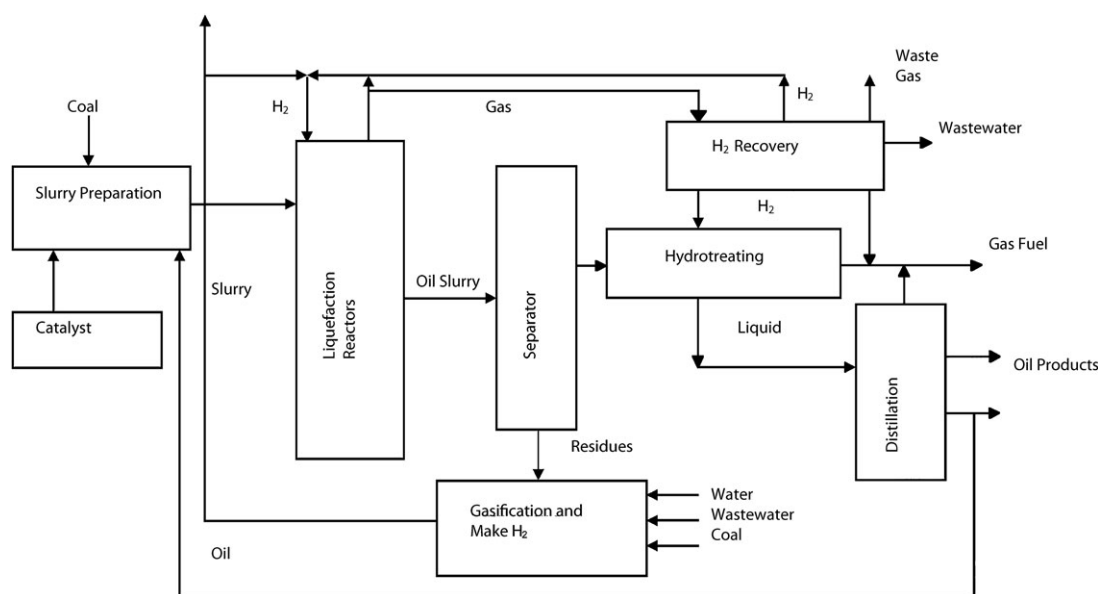
Although technological progress has been made in DCL processes, a number of technical problems that require further research and development include improvement in catalyst and reduction of severity of operating conditions, separation of solids from coal liquids and optimisation of coal liquid refining/distillation and utilisation of the residue (Oster et al., 2009). Unfortunately, no published information on coal liquefaction residue separation is available in open literature in recent years. In this study, the challenge of separating solids from coal liquids is described and reviewed in detail based on available information.

Direct liquefaction residue (DLR) contains solids that include inorganic (catalysts and mineral matter) and organic (unconverted coal and heavy oil) components. Separating solids (mineral matter) from DCL liquid products is necessary for oil recycling. The solid organic residue is used for hydrogen and/or energy generation. Recycling the unreacted coal with catalyst increases catalyst concentration and improves yield, as practiced in the Dow process (Moll and Quarderer, 1979). However, with high levels of

recycling, a separation step is required to prevent mineral matter build-up.

Separation of solids in DCL can be achieved by a range of methods, with hydrocyclones, filters and solvent deashing commonly reported (Leu and Tiller, 1984). For example, the Dow process used hydrocyclones to separate ash from fine catalyst particles. Critical solvent deashing (CSD) was also developed for the vacuum bottoms in the Nippon Brown coal liquefaction process to separate mineral matter and un-reacted coal from coal liquids (Okuma et al., 1998). The CTSL process used filters to separate solids from heavy coal liquids (Comolli et al., 1995).

For the Shenhua SH-I process, an estimated selling price of \$US24 per barrel of fuel is reported (Oster et al., 2009) but this does not include the cost for solids separation, and the residue is gasified without any recycling. However, for the CTSL process, a cost of \$US32 per barrel of fuel has been estimated, which includes the cost of recycling and complete use of the residue. As SH-I is intended to be a commercial process, further work on solids

**Figure 1.** Shenhua commercial process for direct coal liquefaction (Fletcher et al., 2004).

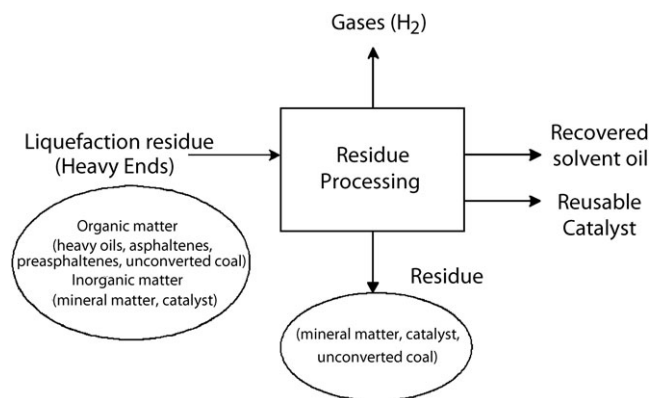


Figure 2. Schematic of DCL residue processing.

separation and recycle is an important consideration (Oster et al., 2009).

The economics and environmental impact of DCL processes depends largely on the end-use of heavy residues after separation. Usually, heavy residues are simply gasified to produce hydrogen for the liquefaction process or to provide heat and power. In the future, however, alternative uses will be required to reduce the carbon footprint of DCL processes. The literature supplies a few possibilities for reducing the carbon footprint, as well as improving the value of heavy residue. These include the production of high-grade carbon materials, such as carbon fibers, nanotubes or graphenes (Zhou et al., 2008) and as pavement asphalt modifier (Yang et al., 2009).

In summary, the objective of DCL residue processing (Figure 2) is to maximise the recovery of recycled liquid by separation of solids, and possible recovery of catalyst, as well as utilisation of the residue. The operations involved in processing of residues are:

- 1 Separation of mineral matter from the unconverted coal and re-usable catalyst.
- 2 Extraction of solvent oil for recycling.
- 3 Conversion of residue for recycled gas or energy production.

This review is focused on separation technologies for recovering of valuable oil and catalyst, while removing ash and unconverted coal from vacuum bottom residue. The residue properties are discussed first.

RESIDUE PROPERTIES

Information on DCL residues in the literature is limited compared with; residues produced in petroleum refining. Even for a given coal, liquefaction temperature, pressure and residence time, catalyst and H-donor solvents are all important factors affecting coal liquefaction performance and residue properties (Cui et al., 2003). Characterisation of the residues is difficult and their properties differ significantly depending on the input coal type, the process (unit operations and operating conditions) and process configurations (combinations of unit operations).

In general, the residues from the DCL process differ from parent coal in having higher concentrations of asphaltenes, pre-asphaltenes and metal compounds. Due to the highly complex nature of coal liquefaction reactions, coal-derived liquids and their residues are generally characterised using solvent solubility (extraction) tests. Oil is usually defined as the hexane soluble material, asphaltenes as hexane insoluble/tetrahydrofuran (THF) soluble and pre-asphaltenes as pyridine soluble/THF insoluble

(THFI). The residues obtained from solvent extraction are commonly characterised by their ultimate and proximate analyses. Residues have also been analysed for boiling point distribution, molecular weight and transport properties such as viscosity.

Table 2 reports some analytical data from the literature for DLRs, and their extracts, from different DCL processes. It includes physical properties of coal liquid residues that the authors have been able to locate. The table includes:

- 1 DLR and THFI materials from Shenhua's 0.1 TPD pilot plant, where the primary coal hydrogenation was carried out in the presence of Fe-based catalyst. The pre-asphaltene fraction (THFI) was difficult to hydrogenate under the primary liquefaction conditions used, but could be hydrogenated to lighter products in the hydrotreater using NiMo/Al₂O₃ catalyst. The results showed lower hydrogen to carbon (H/C) ratios for the THFI solids and higher ash levels compared to the parent liquefaction residue (DLR).
- 2 Winschel and Burke (1989) used vacuum bottoms from the Wilsonville plant runs (250D and E, 251 and 254,) to produce tars in a fluid coker unit and to evaluate them as liquefaction recycle oils.
- 3 Cui et al. (2003) studied the relationship between liquefaction conditions and the properties of THFI residues obtained from the thermal and catalytic hydro liquefaction of two Chinese coals: Yanzhou (YZ) and Fenxi (FX). Volatile matter content was determined using a thermogravimetric analyser ramped up to a temperature of 950°C at 40°C/min and held there for 10 min. Liquefaction temperature and residence time are the main factors that control the properties of the residues. For THFI residues obtained from the catalytic liquefaction, the volatile matter content is higher than the thermal liquefaction for both coals. The addition of catalyst increased the volatile matter content of the residues.

In other study (Martinez et al., 1997) of residue properties reported the kinematic viscosity (4608 cSt at 65°C) and boiling point data for an asphaltenic residue obtained from direct liquefaction of Spanish subbituminous coal.

SEPARATION TECHNOLOGIES

Conventional unit operations typically used for separating solids from liquid products include filtration, hydrocyclones, centrifuges, sedimentation, distillation and solvent extraction methods. Some of these operations have been or may be applied to direct liquefaction residues. Selection of the most suitable solids separation unit operation depends on the nature of the slurry and other process operating conditions. As the compositions and physical properties of slurries produced by the various processes differ, a separation method suited to one process may not be suitable for another. Table 3 lists some solids separation unit operations that have been used for various DCL processes.

Table 4 features several solid-liquid separation operations that have been used in various coal liquefaction processes, and includes operating conditions that were tested at either laboratory and/or pilot scale. Performances of selected separation operations are also reported. Several approaches are possible for separating mineral matter and undissolved organic matter from liquefied coal. Traditional methods such as filtration, centrifugation and hydrocyclones have been used but are not completely satisfactory by themselves. For example in the Dow process, hydrocyclones are employed essentially as thickeners. They can successfully

Table 2. Summary of reported analytical data for coal liquefaction residues*

Sample	Proximate analysis (wt.%)			Ultimate analysis (wt.% daf)						Refs.
	M _{ad}	Ash _d	VM _{daf}	C	H	N	S	O (diff.)	H/C (atomic)	
Shenhua pilot plant direct coal liquefaction residue										
Parent residue produced at 190 bar, 455°C, with dispersed iron catalyst	0.39	11.96	50.81	86.46	5.67	1.06	1.69	5.12	0.79	Li et al. (2008)
Tetrahydrofuran insoluble	0.63	29.8	19.37	84.70	4.64	1.25	4.96	4.45	0.66	Li et al. (2009)
Southern Company's CC-ITSL, vacuum bottoms samples										
Wilsonville (Run 254)—Ohio No. 6 coal		15.4		88.1	6.5	1.0	2.6	1.8	0.88	Winschel and Burke (1989)
Wilsonville (Run 250D, E)—Illinois No. 6		9.8		90.9	6.6	1.0	1.0	0.5	0.87	
Wilsonville (Run 251)—Wyodak coal		31.7		89.3	6.7	1.0	2.9	0.1	0.90	
THFI residue from Chinese bituminous coal (YZ and FX) for 400°C, 60 min										
YZ residue—thermal liquefaction	0.9	3.8	24.2	83.4	4.4	1.9	1.8	8.5	0.63	Cui et al. (2003)
YZ residue—catalytic liquefaction	0.8	12.7	33.0	75.4	3.7	1.4	3.8	15.7	0.58	
FX residue—thermal liquefaction	0.6	9.1	22.2	85.1	4.5	1.5	0.4	8.5	0.63	
FX residue—catalytic liquefaction	0.7	15.1	24.6	86.7	4.6	1.5	0.8	6.4	0.64	

* Ash_d, ash (dry basis); daf, dry ash-free; H/C, hydrogen to carbon atomic ratio; M_{ad}, moisture (air-dried); VM_{daf}, volatile matter (dry ash-free basis); FX, Fenxi coal; YZ, Yanzhou coal.

concentrate dilute suspensions to produce concentrated suspensions (with high solids loading) in the underflow. The underflows can then be further processed in solvent deasphalting unit operations (Moll and Quaderer, 1979).

All two-stage DCL processes require downstream residue separation techniques. The various slurries involved in the processes can have temperatures and pressures from 90 to 450°C and 7–14 bar, respectively, with a typical range of particle diameters of 1–300 µm (GDC, 1975). Coal liquefaction residue is highly viscous and contains high concentrations of solids, including catalysts, unconverted coal particles and mineral matter, which present many challenges for conventional separation operations.

The SRC stage-2 (SRC-II) process includes mineral separation from the coal liquids and involves the use of a rotary pre-coat pressure filter and centrifuges. These could be operated either independently or in combination. The particle size distribution ranged from 1 to 20 µm and continuous solids removal from the filter screen required a filter aid with the rotary pre-coat filter. The centrifuges required high power input to generate enough centrifugal force to separate the particles. Nozzle disk and solid-bowl centrifuges were also used in series to handle the small particle sizes. However, the separation performance could not be guaranteed (GDC, 1975). Initial studies on solid-liquid separation for the SRC-II process were conducted at a pilot-plant facility in Wilsonville, Alabama, USA. A Funda horizontal pressure leaf filter was tested and later modified to a vertical pressure filter (Davis et al., 1980). SRC-II product coal liquid with 0.16% ash was produced from the filtering operation. Pre-coated coarse screens (60 × 60 mesh) were somewhat better than finer screen filters in avoiding problems related to frequent screen blinding, as well as providing improved mechanical strength and ease of fabrication. The rate of blinding was dependent on the type of coal used.

Direct filtration techniques for coal residues (5–10 wt. % solids) have proved to be very difficult. A two-step approach, which involves a combination of pre-thickening of the slurry (using hydrocyclones) before filtration with the use of a filter aid, was suggested as a possibility (Tiller, 1980). Filter area requirements can be substantially reduced by such combinations of unit operations. The process configuration included slurry thickening by the use of hydrocyclone, with the overflow (<0.1% ash) passing through a tank filter (with filter aid) and the underflow passing through a pressurised drum filter (Tiller and Webb, 1980).

A stand-alone hydrocyclone was tested for its performance in separating a coal slurry feed containing 1–6 wt. % solids (Tiller and Webb, 1980). This resulted in an overflow with ~1 wt. % ash and an underflow with 23 wt. % ash. However, the performance was not acceptable (high ash levels in overflow) for the Exxon donor solvent (EDS) process, which subsequently utilised a combination of vacuum distillation and flexi coking operations (Fiocco, 1979). Similarly, hydrocyclones could not be used in the SRC-II process at Wilsonville due to the low solids concentration in the underflow.

For good solid-liquid separation, the viscosity of the slurry has to be reduced, either by increasing the temperature and/or by dilution of the residue stream with an extraction solvent. Higher temperatures (260–400°C) cause vaporisation of the solvent, although this can be prevented by increasing the operating pressure up to 14 bar (GDC, 1975). Successful continuous separation under high pressure using a rotary pre-coat filter and centrifuge was difficult to achieve, as it involved discharge of solids from high to atmospheric pressure (GDC, 1975). However, a high viscosity Sier-Bath screw pump was used to manage the pressure seal by allowing solids levels to be maintained in a long standpipe. Pressurised rotary drum pre-coat filters were also used

Table 3. Solids separation operations used in various direct coal liquefaction processes*

DCL processes	Solids separation operation
CSF	Hydrocyclones in series
H-Coal	Hydrocyclones followed by vacuum distillation
Dow process	Hydrocyclones followed by solvent deasphalter
Wilsonville	Kerr-McGee—critical solvent deashing (CSD)
CTSL/CC-ITSL	
LSE	Vertical leaf pressure filter
SRC-II	Rotary pressure pre-coat filter/centrifuges
BCL	Solvent deashing
COED	Pressurised rotary drum pre-coat filters
EDS	Vacuum distillation followed by flexi-coking

* Source: see Abbreviations list.

Table 4. Examples of solid–liquid separation techniques used for processing heavy ends from direct coal liquefaction

Technology	Features	Examples	Process conditions	Outcome	Refs.
Hydrocyclones (liquid cyclones)	Advantages	Cresap Pilot Plant (Consolidation Coal Company) comparative study of: 1. hydrocyclone; 2. continuous pre-coat filter; 3. continuous pressurised centrifuge	Temperature 260–315°C and 10 bar	Two-stage hydrocyclone most successful with 90% recovery of coal extract containing <1% ash	Phinney (1974)
	Simple construction High efficiency <1 wt.% solids in overflow Good operability	Study by Shell Research Laboratory, Amsterdam on asphaltenes from residual oil in pilot and semi-commercial units using hydrocyclones in either series or parallel arrangements	Four parallel cyclones of 50 mm or single cyclone of 83 mm	80–90% of the asphaltenes separated	Leu and Tiller (1984)
	Low operating cost Disadvantages	SRC-I Wilsonville coal liquefaction sample	10, 25 and 50 mm diameter cyclones used with anti-solvent to promote separation	Solid removal efficiency of 70–80% with <1 wt.% ash in overflow	Fiocco (1979)
	Requires low slurry viscosity 5–20% loss of liquid in underflow	CSF using a two-stage cyclone of 45 and 90 L/min capacity and processing slurries with 5–20 wt.% solids	Temperature 260–315°C and 6–10 bar with 1–300 µm diameter particles	Two-third of the feed solids could be recovered in the underflow	GDC (1975), Katz et al. (1974)
Rotary pressure pre-coat filtration	Separation efficiency low for particles <10 µm Low tolerance to feed variations for given size unit	Dow's coal liquefaction process with solids separation using hydrocyclones	10 mm diameter hydro-cyclone with feed rates of 0.08 kg/s at 250°C and ΔP of 7.6 bar; 6 wt.% ash in feed	Feed solids separated into overflow (<3 wt.% ash) and underflow (11 wt.% ash)	Moll and Quaderer (1979)
	Requires multiple cyclone arrangements (increased cost) for achieving high separation efficiency (or low cut size)	Mineral and undissolved solids separation from coal liquids in the SRC-I process using different filters: Funda horizontal filter (5–10 m ²); US Filter Corp. (area of 81 m ²); Goslin-Birmingham drum filter	Feed slurry 30 wt.% SRC product and 4–8 wt.% insoluble particles of 3–10 µm, 260–400°C and 6.8–14 bar using a Goslin-Birmingham drum filter of 3.7 and 7.4 m ² filtering area	Filtration rates 245–985 kg/h/m ² with ash levels 0.2 wt.% in the SRC product	GDC (1975)
	Advantages High output per unit area Good separation efficiency Handles slurry with high solids loading Particle separation with size <10 µm				

(Continued)

Table 4. (Continued)

Technology	Features	Examples	Process conditions	Outcome	Refs.
	Disadvantages	Study on solid-liquid separation at a 36 TPD pilot plant processing product slurry obtained from the COED process oil (FMC Corp.)	5–10 wt.% solids in COED process oil	Filtered oil with <0.1 wt.% solids obtained at rates of 0.34–0.44 m ³ /h/m ²	Leu and Tiller (1984), Katz et al. (1974)
	High capital and operating cost Mechanical problems during operation and cleaning Difficult for continuous solids discharge Requires low slurry viscosity	Processing of H-Coal product slurry stream using a Goslin-Birmingham continuous filter with pre-coat	0.4 m ² of filtering surface area available	Ash levels of 0.04–0.06 wt.% produced	
	High consumption of filter aid and cake disposal issue High solvent loss up to 15 wt.% in cake				
Centrifugal sedimentation (solid bowl/nozzle-disk centrifuge)	Advantages	SRC product (Wilsonville pilot plant) used as a feed for testing separation performance of a solid bowl centrifuge	343°C and 10 bar	Coal liquid feed containing 5 wt.% solids is separated into the SRC oil rich stream with 0.2 wt.% ash	Leu and Tiller (1984)
Critical solvent deashing (CSD; Kellogg Brown & Root, Inc.)	High throughput Separation of particle size <10 µm				
	Disadvantages Mechanical abrasion High capital cost				
	Advantages	CSD unit installed in SRC process, Wilsonville	Uses proprietary solvents (including pentane, hexane, etc.) under critical point	78 wt.% recovery of SRC product with 0.08 wt.% ash, solid residue stream of 50–65% ash and unconverted coal	Adams et al. (1979) Penner (1980)
	Wide range of feedstocks with high solids loading High throughput Ash <0.1 wt.% in deashed coal and SRC solvent	Dow's 23 kg/h counter current solvent deasphalter to separate ash and unconverted coal from product oil	Feed stream containing 15–16 wt.% ash treated with mixed solvent rich in hexane	Ash concentration in top product <0.02 wt.% with 40 wt.% in the bottom residue stream	Moll and Quaderer (1979)
	Powdered solid product and ease of separation of solids	Coal liquefaction bottoms stream obtained from the 1st stage hydrogenation step of the BCL process (two-stage process) using Morwell and Yallourn coals in a 50 TPD pilot plant (Victoria)	Process conditions of 200–290°C and 40–50 bar used to separate ash and heavy pre-asphaltenes using toluene	Deasphalted oil obtained with ash levels <3000 ppm	Okuma et al. (1997)
	Disadvantages				
	Loss of solvent in the solids residue (15 wt.% of feed) High pressure				

by the FMC Corporation in its 36 TPD pilot plant in Princeton (New Jersey) to demonstrate that coal oil slurry with 10 wt.% solids could be filtered to less than 0.1 wt.% solids at filtration rates of 0.34–0.44 m³/h/m² (Leu and Tiller, 1984).

The H-Coal process developed by HTI used a Goslin–Birmingham continuous pressure pre-coat drum filter (0.44 m² area) at their Lawrenceville pilot plant to produce 0.04–0.06 wt.% ash in recycled oil. However, the filter was poorly designed and difficult to access for cleaning. The SRC-II pilot plant at Tacoma, Washington, used two similar filters with filtering areas of 3.7 and 7.4 m². The operating conditions used were 14 bar and 400°C. The feed was 30 wt.% SRC slurry containing 4–8 wt.% insoluble particles (ash) of 3–10 µm size. The filtration temperature was 260°C with a shell pressure of 8–11 bar.

The Dow Process (Moll and Quaderer, 1979) used a hydrocyclone to concentrate solids in coal liquefaction product streams. Operating data on a 10 mm hydrocyclone unit with feed rates of 0.079 kg/s have been presented, claiming superior performance over other separation technologies. An operating temperature of 250°C with a pressure drop of 7.6 bar was used. A hydrocyclone feed with ash levels of 6 wt.% was separated to give 3 wt.% ash in the overflow and 11 wt.% in the underflow. The underflow was then fed to a solvent deasphalter unit. The deasphalter unit could handle feed streams containing 11 wt.% ash and produce a residue containing ~65 wt.% total solids.

The Cresap pilot plant used three separation operations, including hydrocyclone, continuous pre-coat filter and continuous pressurised centrifuge at 315°C and 10 bar pressure. However, a two-stage hydrocyclone was found to be more effective, with 90% recovery of the coal extract with <1% ash content (Phinney, 1974). Shell Research Laboratories used hydrocyclones in parallel and series arrangements to remove 80–90% of asphaltenes from residual fuel oils (Leu and Tiller, 1984).

Gutterman (1994) and Curtis et al. (1995) used a different approach to the processing of direct liquefaction residue from the atmospheric column bottoms using black thunder sub-bituminous coal. Also known as the Foster Wheeler's Asphalt Coking Technology (ASCOT) process, it included solvent deasphalting and delayed coking units to produce a distillate fraction and a smaller residue fraction. The tests were conducted on a batch-scale deasphalting unit (16 L pressure vessel) and a 100 mm delayed coking unit facility. The ASCOT process produced an average liquid yield of 62.4 wt.%, which was greater than the combined liquid yield from the vacuum tower and CC-ITSL CSD process (58.6 wt.%), as well as the stand-alone delayed coking unit (50 wt.%). The test produced liquid products with low ash (<0.02 wt.%) and metallic content (Ni and V <1 ppm; Cu and Na <4 ppm; Fe 61 ppm).

CSD is a relatively new process developed to separate mineral matter and un-reacted coal from coal liquids (Davis et al., 1980). It works on the principle of selective extraction of coal liquefaction products using organic solvent near its critical temperature and pressure. The initial CSD process run achieved ash levels of 0.06% in the SRC-II product with a loss of solvent to the products of less than 1% of the feed. Kerr–McGee developed a three-stage CSD process which was used to extract deashed coal as a main product in the SRC-II process. Since the installation of CSD unit at the Wilsonville test facility, the CSD unit was used in all test runs on CC-ITSL configuration. Dow has successfully implemented CSD in combination with hydrocyclones for solids separation.

CeraMemCorporation (1995) investigated a DLR separation operation involving a cross-flow filtration concept. Sonically assisted cross-flow filtration was performed on the residue sam-

ple obtained from the CC-ITSL (Wilsonville run 260). Pulsed sound increased the filtration rate by 600-fold over simple filtration (Slomka, 1994). Other small-scale studies involved inducing a high-voltage electric field over the cross-section of a tubular filter, which improved separation performance (Lee et al., 1980).

CONCLUSIONS

There is general consensus in the literature that one of the most important problems to be resolved in the commercial development of DCL processes is the separation of solids from valuable coal liquids and utilisation of the residue stream.

The compositions and physical properties of residues are dependent on the process type, and a separation operation well suited to one process may not be so for another. Extensive laboratory and pilot-plant programs in the United States of America, Japan, Germany, China and Australia have improved our understanding of solids separation operations that can be applied to coal liquefaction. Unit operations such as filtration, centrifugation, hydrocyclone and solvent deashing were reviewed for their ability to separate mineral matter and unconverted organics from coal liquefaction residues. Individual operations used have not been completely satisfactory, and a better overall result is obtained when they are used in combination. The combination of hydrocyclones and CSD appears to show the greatest promise as a two-stage process.

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