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CALI 07.09.79 H(9-A1) 02132 J/47 H09 CHEVRON RESEARCH CO \*US 4358-359 26.09.80-US-191454 (+073156) (09.11.82) C10g-01/06 Two stage coal liquefaction - using process-derived solvent having DETAILS low heptane-insolubles content

C.i.p. of 4255248

A coal liquefaction process is effected by (a) slurrying the subdivided coal in a solvent (I), (b) passing the slurry, with added H2, through a dissolving zone to produce a first effluent (A), (b) catalytically hydrocracking (A) to produce a second effluent (B), (c) sepg. residual coal from (B) to produce a solids-lean liq. (C), (d) cooling at least a portion of (C) to precipitate substantially all the heptane-insoluble constituents, (e) sepg. the precipitate from the liquid, and (f) recycling the heptane-insolubles-free liquid for use as in step (a).

## ADVANTAGES

The use of costly anti-solvents for heptane-insolubles removal is eliminated. The process vields extremely clean normally liquid products, those boiling above 4C having unusually low s.g., an S content of < 0.1 wt. % and an N content of < 0.5 wt. %.

The (I): coal wt. ratio is pref. 1-2:1. The dissolving zone is pref. operated at 425-450 (esp. 440-450) °C, 35-680 (esp. 100-170) atm. with an H<sub>3</sub> rate of 355-3550 (esp. 380-1750) 1/1 and with a residence time of 0.1-2 (esp. 0.25-2) hrs. The hydrocracking zone is pref. operated at 340-400 °C, 70-205 atm., an H<sub>2</sub> rate of 355-3550 1/1 and a slurry LHSV of 0.1-2. Sepn. in steps (c) and (e) may be affected by filtration,

+ boiling fraction. The cooling is pref. to 16-95 °C to reduce the heptane-insoluble level in the lig. from 2-5 wt. % to < 1 (esp. < 0.5) wt. % (7pp 920)

The portion of (C) subjected to cooling is pref. a 200 °C

gravity, hydrocloning and/or centrifuging.

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H09

Coal liquefaction by solvent treatment and hydrocracking - with removal of heptane insolubles by cooling the lig. prod.

Coal liquefaction is carried out by (a) slurrying particulate coal in a solvent; (b) passing the slurry with H2 through a dissolution zone which contains no externally supplied catalyst or contact particles and is operated at 425-480°C; (c) subjecting the effluent to catalytic hydrocracking at 340-400 °C and 70-205 atm; (d) cooling at least part of the liq. product to pate, the heptane insolubles (HI); and (e) recycling at least part of the HI-free liq. product to step (a) as solvent.

## **ADVANTAGES**

The process yields a high-quality recycle solvent fraction without the use of expensive antisolvents for HI pptn.

## DETAILS

Step (b) is pref. effected at 440-450 °C and 35-680 (esp. 100-170) atm, with a solvent:coal wt. ratio of 0.5-5:1 (esp. 1-2:1) and an H2:slurry vol. ratio of 355-3550:1 (esp. 380-1480:1). The residence time is pref. 0.1-2 (esp. 0.25-1) hr. H(9-A1).

(19pp367).

BE-884558

Step (c) is pref. effected at a temp, 25-140°C below the temp, in the dissolution zone and approx, the same pressure

with an LHSV of 0.1-2 (esp. 0.2-0.5), using a fixed bed of

Co/Mo/Al<sub>2</sub>O<sub>3</sub>, Ni/Mo/Al<sub>2</sub>O<sub>3</sub> or Ni/W/Al<sub>2</sub>O<sub>3</sub> catalyst. The hydrocracked effluent is pref, subjected to gravity seen. at 150-205 °C and 1-70 atm. to remove solids, giving

a liq. product contg. 2-5 wt.% HI and 0.1-0.5% coal residues. The liq. product (or a 200°C + fraction thereof) is then

cooled to 16-95 °C and filtered or centrifuged to remove HI.