

8. Desulfurization

1973

8.1 55719c Pilot plant for solvent refining of coal. Anon. (USA). Mining Congr. J. 1973. 59(1), 68-69 (Eng). A flowsheet is given for extn. of S from coal. The coal is dissolved in a coal-based solvent at 1000 psi and 427° in the presence of H. After the pressure is reduced, the coal soln. is filtered and the filtrate evapd. to remove solvent for recycling and obtain a refined coal. The S is recovered from the gases formed during dissoln.

1972

8.2 143235k Removal of sulfur from coal by air oxidation at 350-450°. Sinha, R. K.; Walker, P. L., Jr. (Dep. Mater. Sci., Pennsylvania State Univ., University Park, Pa.). Fuel 1972, 51(2), 125-9 (Eng). S removal from 7 U.S. coals (total S, 3-7 wt. %) by low-temp. oxidn. was studied. Approx. 50% of the total S was pyritic. Oxidn. was carried out largely at 350, 400, and 450°. Temp. up to 450° was a prime factor in detg. the extent of desulfurization. The efficiency of removal was controlled by diffusion of O into coal and its pyrite. In some cases >90% of the pyritic S could be removed in 10 min at 450°. The rate of removal was unaffected by increase of SO₂ in the air stream to at least 10%, hence the level of SO₂ could be increased for H₂SO₄ by successive recycling of effluent gases over fresh coal charges. The coal calorific value and volatile matter decreased during this desulfurization treatment, but this loss can be minimized by feeding the desulfurized coal at 450° directly to the boiler. The process could be made self-sustaining by proper control of the coal/air ratio.

8.3 94541h Selection of the optimum amount of Raney nickel catalyst additive for the desulfurization of western Donets Basin coals. Samoilenko, G. E.; Gerasimenko, L. G.; Vorontsova, Z. V.; Savin, M. I. (USSR). Met. Koksokhim. 1972, No. 32, 69-71 (Russ). The coals were heated at 250° for 0.5, 1.0, 1.5, and 2.0 hr in the presence of 1, 2, 3, and 4% catalyst by wt. The coals studied contained 1.65% S. Equations are given for calcn. of the most effective amt. of catalyst and the optimum final S content. Most of the decrease in the S content occurred during the 1st hr of heat treatment.

8.4 46055k Desulfurization of coals and chars by treatment in various atmospheres between 400 and 600°. Sinha, R. K.; Walker, P. L., Jr. (Dep. Mater. Sci., Pennsylvania State Univ., University Park, Pa.). Fuel 1972, 51(4), 329-31 (Eng). Desulfurization of 15 U.S. coals ranging from anthracite to sub-bituminous and of 5 high-temp. chars by CO and other gases and

gas mixts. between 400 and 600° was studied. The S content of the parent coals was 3.0-7.3 and that of the chars 1.3-3.8 wt. %. The order of efficiency of the desulfurizing gas is air > steam-CO mixt. > CO > N.

8.5 142164v Production of low-sulfur boiler fuel by two-stage combustion. Application of carbon dioxide acceptor process. Curran, G. P.; Fink, C. E.; Gorin, E. (Consolidation Coal Co., Library, Pa.). Air Pollut. Contr. Off. (U. S.) Publ. 1972, AP-109, III-1, 12 pp. (Eng). A modification of the CO₂ acceptor process is presented as a method for producing low-S boiler fuels, i.e. low-S char and low-S producer gas. Particular emphasis is given to the total gasification of eastern coals for the production of low-S producer gas which could be utilized for the generation of clean power in new combined-cycle plants. Exptl. data are given on acceptor life under projected operating conditions.

8.6 142154s Clean power systems using fluidized-bed combustion. Squires, A. M. (City Coll., City Univ. New York, New York, N.Y.). Air Pollut. Contr. Off. (U.S.) Publ. 1972, AP-109, IV-1, 10 pp. (Eng). Systems for clean power from coal involve the combustion of coal at a high pressure in the presence of a desulfurizing agent and the generation of power by a combination of gas-turbine and steam-turbine cycles. Three combustion technol. approaches, e.g. complete combustion, partial combustion, and carbonization, for use in the above-mentioned combination are discussed and the power cycle arrangements suited for each combustion approach considered.

8.7 142150n Desulfurization of coal. Meyers, R. A.; Hamersma, J. W.; Land, J. S.; Kraft, M. L. (TRW Syst. Group, Redondo Beach, Calif.). Science 1972, 177(4055), 1187-8 (Eng). Coal combustion is the largest single source of S oxide pollution. Treatment of some major representative U. S. coals with aq. Fe³⁺ soln. removes 40-75% of the S content through near quant. oxidn. of the pyritic S contained in the coal matrix. Elemental S and iron sulfate are recovered as products of the reaction. Engineering assessment indicates that the system, when fully developed, offers high potential for the economic abatement of S oxide pollution.

1971

8.8 129710m Chemical removal of nitrogen and organic sulfur from coal. Meyers, Robert A.; Land, J. S.; Flegal, C. A. (TRW Syst. Group, Redondo Beach, Calif.). U. S. Nat. Tech. Inform. Serv., PB Rep. 1971, No. 204863, 62 pp. (Eng). Avail. NTIS. From Govt. Rep. Announce. (U.S.) 1972, 72(3), 189. Techniques for extg. org. S and N compds. from coal without

significantly altering the remainder of the coal matrix were tested in the lab. The concept for chem. removal of org. S and N is feasible. Weak org. acids remove 45-80% of the org. S from the coals investigated depending on process conditions and the specific coal leached. PhNO_2 appears to be the most efficient solvent for the removal of org. S. Aq. caustic removes small amts. of org. S from some coals, but none from others. Particle size (--200 mesh to 1/2-inch) has no discernable effect on the removal of org. S by PhNO_2 . The leaching solvent, PhNO_2 , may be recycled at least 3 times to remove org. S from coal. Weak org. acids and strong inorg. acids appear to remove N from the coals tested in this program according to the Dumas anal. method, but no N is removed according to the Kjeldahl method. Weak org. acids for removal of org. S from coal may be readily adapted to engineering process schemes.

8.9 113711m Sulfur behavior and sequestering of sulfur compounds during coal carbonization, gasification, and combustion. Vestal, Marvin L.; Day, Allan G., III; Snyderman, J. S.; Fergusson, Gordon J.; Lampe, F. W. (Sci. Res. Instrum. Corp., Baltimore, Md.). U. S. Nat. Tech. Inform. Serv., PB Rep. 1971, No. 211481, 136 pp. (Eng). Avail. NTIS. From Govt. Rep. Announce. (U.S.) 1972, 72(20), 224. The theoretical, exptl., and practical problems assocd. with the desulfurization of coal were studied by using nonisothermal kinetic methods. The nonisothermal kinetics was studied of the desulfurization and gasification of 5 bituminous coals, lignite, anthracite, and pyrite, under reducing and oxidizing atms. An attempt was made to identify the types of S bonding assocd. with the removal of S. The nonisothermal kinetics was studied of the use of both CaO and dolomite to remove H_2S under both reducing and oxidizing conditions. Reactions of H_2S with Fe, Fe sulfide, and various coal chars also were investigated.

8.10 142179d Pyritic sulfur removal from coal using solutions containing ferric ions. Meyers, Robert Allen (TRW Inc.) Ger. Offen. 2,207,491 (Cl. C 10 1), 24 Aug 1972, US Appl. 116,262, 17 Feb 1971; 20 pp. The above removal is carried out together with addnl. extn. with an org. solvent. An FeCl_3 soln. is used, and the spent Fe contg. solns. can be regenerated. After treatment with the Fe^{3+} soln., the coal is filtered off, washed repeatedly with vacuum rotary filtering between, dried at 100° , the S is evapd. by heating to $\sim 230^\circ$, and excess FeCl_3 soln. is transferred to the initial stage. Addnl. S is removed by treatment with a S solvent at $80-200^\circ$. Of many types of coal, Indiana No. V showed that up to 93% of the pyrite S could be removed during a 2-hr treatment with a 0.5M FeCl_3 soln., which could be further improved by addnl. solvent extn. with p-cresol at 200° . The absence of high pressures and temps. for this process, is also of advantage.

8.11 74585k Low-sulfur fuel oil from coal. Akhtar, Sayeed; Friedman, Sam; Yavorsky, Paul M. (Bur. Mines, Washington, D.C.). U.S. Nat. Tech. Inform. Serv., PB Rep. 1971, No. 203889, 13 pp. (Eng). Avail. NTIS. From Govt. Rep. Announce. (U.S.) 1971, 72(24), 182. A high-S bituminous coal suspended in coal tar was hydrodesulfurized by continuous processing through a fixed bed of pelletized Co molybdate on alumina catalyst, under conditions of highly turbulent flow of H to prevent obstruction of the flow of fluids and to promote catalytic contact. High yields of low-S fuel oil were obtained. The feed contained 30% coal contg. 3.4% S suspended in tar contg. 0.6% S. For process conditions of 4000 psig and 450°, the yield of whole liquefied product was 94% of the whole feed; the product had 9% benzene-insol. residues and only 0.3% S. Most of the sulfur was in the insol. residues and was organic. By sepg. the insol. residues, the whole product gave a 91% yield of benzene-sol. fuel oil having only 0.09% S. For the milder conditions of 2000 psig and 450°, the whole liquefied product was 93% of the feed and had 13% insol. residues and 0.4% sulfur. By separating the residues, this product gave an 87% yield of fuel oil having only 0.14% S. Part of the sepd. product oil can be recycled to suspend the feed coal instead of using the tar. Thus, the net product oil for fuel use can be derived entirely from coal and is expected to have S at least as low as reported above. The work presents a method for providing large amts. of low-S fuel.

1970

8.12 143261r Hydrogen processing of coal and the kinetics of desulfurization. Vestal, Marvin L.; Essenhig, Robert H.; Johnston, William Harper (Sci. Res. Instrum. Corp., Baltimore, Md.). Amer. Chem. Soc., Div. Fuel Chem., Prepr. 1970, 14(4) (Pt. 2), 84-100 (Eng). Kinetic studies were made on the desulfurization reactions during the pyrolysis and gasification of bituminous coal in H. The powerful method of nonisothermal measurement of Juntgen (1964) was extended and applied successfully to account for better than 95% of the S. The activation energies and corresponding frequency factors were measured for 5 desulfurization reactions which account for the desulfurization in 10 different coals from Illinois, Ohio, Pennsylvania, Kentucky, and Maryland. These data are applied to the establishment of conditions which must be met in order to accomplish efficient and therefore economic desulfurization during the H processing of coal. A continuous process scheme is proposed which satisfies these conditions. This scheme is analyzed by way of a SANKEY energy flow diagram. The conclusions of this anal. are discussed including the major result that the material and energy flows are acceptable and compatible with realistic reactor temps. A specific reactor design which appears attractive for this use is discussed.

8.13 101435d Effect of a Raney nickel catalyst on the desulfurization of Donetsk coals. Samoilenko, G. E.; Vorontsova, Z. V.; Savin, M. I. (USSR). Met. Koksokhim. 1970, No. 22, 15-18 (Russ). Raney Ni was used to desulfurize org. and inorg. compds. The action of Ni catalysts on coal was studied by heating at 250° and by boiling in water. The amt. of catalyst on heating was 2.5% and at boiling 5 and 10%. The catalyst reduced the S content in most coals from 7 to 26% and in some from 33 to 40%. Soft coal lost 54.9%; boiling with 10% catalyst removed 23-36% S. Since the catalyst contained adsorbed H, the pyrite reaction can be carried to Ni_3S_2 , then to Ni.

8.14 153637m Chemistry and kinetics of the hydrosulfurization of coal. Vestal, Marvin L.; Johnston, William H. (Sci. Res. Instrum. Corp., Baltimore, Md.). Amer. Chem. Soc., Div. Fuel Chem., Prepr. 1970, 14(1), 1-11 (Eng.). The kinetics of desulfurization of 10 different bituminous coals during gasification in H are accounted for satisfactorily by 5 reaction systems whose activation energies and corresponding frequency factors are measured by the theoretical extension and exptl. application of the Juntgen nonisothermal method. The kinetics of a series of back reactions are also measured including the scavenging of H_2S by CaO. The reactions of desulfurization are: (1) and (2) two different low-temp., irreversible pyrolysis reactions to produce H_2S ; (3) the reaction of Fe pyrites with H to form FeS; (4) the reversible redn. of FeS to Fe; and (5) the complex and partially reversible redn. of high-temp. C-bound S, which occurs in all coals studied and could be simulated with the reaction product of H_2S with mineral-free charcoal. The coals contained 1-5% S and originated from Illinois, Ohio, Pennsylvania, Kentucky and Maryland. The applications of these desulfurization reactions to processes for environmental pollution control are discussed.

8.15 149602r Hydrotreating of hydrocarbonaceous liquids with carbon monoxide-containing gas. Vernon, Lonnie W.; Pennington, Robert E. (Esso Research and Engineering Co.) U.S. 3,719,588 (Cl. 208/209; C 10g), 06 Mar 1973, Appl. 84,089, 26 Oct 1970; t pp. Hydrocarbon oils contg. combined N and S and b. 350-1000°F are hydrotreated in the presence of CO-sensitive hydrogenation catalysts with a gas contg. ≤ 2 volume % CO and 1-20 moles of steam/mole CO. The gas may also contain ≤ 96 vol. % H. Preferably, a coal-derived liq. is hydrotreated in the presence of Co molybdate catalyst with a gas contg. 3 mole % CO and 10 mole % H_2O at 650-800°F, 500-2500 psig, a treating gas-liq. ratio of 3,000-12,000 ft^3/bbl , and the liq. space velocity of 0.5-2.0 hr^{-1} . Thus, crude creosote oil b. 4000-700°F was charged with a 1:1 CO-steam mixt. to a reactor contg.

a Co molybdate catalyst at 750°F and 1000 psig, resulting in 95% desulfurization and 65% denitrogenation of the feedstock.

8.16 51078d Forming a low-sulfur residual fuel oil from a high-sulfur coal. Jacobs, Harry E.; Worrell, George R. (Atlantic Richfield Co.) U.S. 3,663,420 (Cl. 208-8; C 10g), 16 May 1972, Appl. 80,673, 14 Oct 1970; 4 pp. Coal is hydrogenated in the absence of externally supplied contact particles at 500°F to partially gasify and liquify the coal. The solid particles (ash, char, etc.) are sep'd. from the resulting liq. at a min. temp. of 200°F below the hydrogenation temp. The resulting liq. product is useful as a low-S residual fuel oil.

1969

8.17 6349c Group composition of sulfur-organic compounds in petroleum and coal distillates. Nikudina, S. E.; Baranskii, A. D.; Kudashov, F. I.; Serova, N. A. (USSR). Izv. Nauch.-Issled. Inst. Nefte-Uglekhim. Sin. Irkutsk. Univ. 1969, 11(Pt.1), 88-90 (Russ). Total S, sulfide S, mercaptan S, disulfide S, and elementary S were det'd. in the light fractions of the low-temp. Tceremhovski coal tar and in the fractions of the Markovski crude oil having a b.p. $\leq 200^\circ$. The amt. of mercaptan S in the crude oil fractions was 0.28-0.44 wt. % compared to 0.010-0.007 in the coal tar distillates. In both, the amt. of mercaptan S increases with increasing fraction b.p. Disulfide S in the crude oil fractions is insignificant, but in the coal products it is 0.08-0.12 wt. %. Crude oil products anal. methods can be used to det. a group compn. of org. S in coal distillates which have been previously treated to remove acidic and alk. compds.

8.18 6350w Genetic features of the sulfur Karantsaisk coals. Baranskii, A. D.; Fisenko, N. N. (USSR). Izv. Nauch.-Issled. Inst. Nefte-Uglekhim. Sin. Irkutsk. Univ. 1969, 11(Pt. 1), 86-7 (Russ). In a study of 31 samples most of which contain org. S and the rest mineral S, the amt. of org. S increases with increasing coal-layer depth, due to the penetration of H₂S-contg. water into the tectonic cracks. The amt. of active S groups was exam'd. in natural coals, residual coals, and in the coal solvent exts.

8.19 79817y Behavior of sulfur compounds in coal during treatment with a solution of sodium in ammonia. Angelova, Gergina; Lazarov, Lazar; Kovacheva, V. (Inst. Allg. Anorg. Chem., Sofia, Bulg.). Brennst.-Chem. 1969, 50(1), 11-14 (Ger.). Samples of coal with varying S content and rank were treated with Na in NH₃ at -35 to -40° under dry N for 4-5 hrs., using a coal-to-Na ratio of 1:1. Org. Na compds. and excess Na were then decomp'd. by the addn. of dry NH₄Cl and moist ether. The

mixt. was acidified with HCl and filtered. The residue was washed until free of Cl ion, dried in vacuo at +70°, and analyzed. Na₂S and H₂S were formed. The treatment reduced the S present as pyrite as well as the amt. of org. S present. The amt. of decrease varied depending on the origin of the coal sample. However, the Na treatment increased the no. of SH groups present, probably by cleavage of sulfide and disulfide bonds. Coal with a high content of reactive C-S-C bonds exhibited very good coking properties. The unreactive S was thought to be contained in heterocyclic compds. The behavior of the S compds. was compared to that of O compds in the coal.

8.20 123747t Catalytic hydrogenation of coal. Krichko, A. A.; Titova, T. A.; Konyashina, R. A.; Filippov, B. S.; Nikiforova, T. S.; Bronovets, T. M. U.S.S.R. 257,484 (Cl C 10b), 20 Nov 1969, Appl. 14 Aug 1968; From Otkrytiya, Izobret., Prom. Obraztzy, Tovarnye Znaki 1969, 46(36), 24. Coal and paste-forming agents underwent catalytic hydrogenation at high temp. and H pressure. To decrease H consumption, a mixt. of low-S coal with a S-contg. feed (high-S or topped petroleums) was hydrogenated in the presence of initiating additives, such as 0.1-10% inhibitors [?] of radical polymn., at 400-50° and 50-150 atm.

1968

8.21 79292y Electrolytic reduction of lignin and lignites in alkaline solutions. Itoh, Hironori; Ishii, Tadao; Takeya, Gen (Hokkaido Univ., Sapporo, Japan). Hokkaido Daigaku Kogakubu Kenkyu Hokoku 1968, (49), 141-53 (Japan). Soluble lignin and lignite were made in aq. alkali. Powd. H₂SO₄ lignin (I), Tokachi lignite (II), and Sohya coal (III) were suspended in a N LiOH or NaOH soln., which served as electrolytes. The cathode and anode consisted of Pb and Cu plates, resp. The applied c.d. of the d.c. used was 0.03 amp./cm.² The temp. in the cathode vessel contg. the sample suspension was 55 ± 2°. After electrolytic redn. for 10 hrs., the content of the cathode vessel was filtered and divided into the residues (IV) and the alkali-sol. part. The latter was sepd. into a ppt. (V) which appeared when acidified by addn. of HCl and the MeCOEt ext. (VI) of the acidic soln. In order to det. the effect of the electrolytic treatment, the powd. samples were dissolved in an alk. soln. under the same conditions as in the electrolytic treatment but with no elec. current. On electrolytic redn. of lignin and lignite, the yields of materials dissolved in aq. alkali increased to 69.7, 28.4, and 13.8% from 59.0, 19.3, and 2.1% from I, II, and III, resp. Material balances in IV, V, and VI are presented for I, II, and III. When V was subjected to electrolytic redn., the N NaOH-sol. part (3.5%) increased to 43.6% in I and from 10.9 to 74.3% in II. Also, the ir-absorption bands at 1700 cm.⁻¹ were more intensified by the electrolytic treatment.

8.22 101203b Recovery of sulfur values from sulfur-bearing carbonaceous materials. Lefrancois, Philip A.; Barclay, Kenneth M.; Van Hook, James P. (Pullman Inc.) U.S. 3,567,377 (Cl. 28-181; C 01b, C 10g), 02 Mar 1971, Appl. 12 Aug 1968; 7 pp. A S-contg. carbonaceous material, e.g. bituminous coal, is contacted with molten Na_2CO_3 at 1550-2000°F and 200-500 psia. The S compds. in the material are thereby converted to Na sulfide which remains in the melt. The Na sulfide-contg. melt is then dissolved in aq. NaHCO_3 soln. to form an aq. soln. contg. Na sulfide, Na_2CO_3 , and NaHCO_3 . The soln. is filtered, and the filtrate is contacted at 50-150°F and atm. -100 psia with CO_2 in a 1st carbonation zone, the soln. being withdrawn before a substantial amt. of gaseous H_2S is formed. The soln. is then carbonated in a 2nd zone in which a major part of the Na sulfide is converted to gaseous H_2S . The H_2S is withdrawn as product, suitable for conversion to S or H_2SO_4 by usual methods. The soln. from the 2nd carbonation zone contains pptd. NaHCO_3 , part of which is sepd. before recycling the soln. to the 1st carbonation zone.

8.23 68344c Hydrogen from sulfur-containing hydrocarbon fuels. Pelczarski, Eugene A.; Karnavas, James A. (Black, Sivalls and Bryson, Inc.) Ger. Offen. 1,955,115 (Cl. C 01b, C 02b), 18 Jun 1970, US Appl. 01 Nov 1968; 16 pp. H is prepd. from sulfurous, coal, lignites, or peats of low calorific value. The pulverized fuel reacts in a molten iron bath with O and steam. The bath surface is covered with a layer of limestone. Slag and CaS are produced in the layer and other gases escape through it. Slag is continuously withdrawn and fresh limestone used.

1967

8.24 5269v Electrochemical reduction of coal. Sternberg, Heinz W. (U. S. Bur. of Mines, Pittsburgh, Pa.). Symp. Sci. Technol. Coal 1967, 168-74 (Eng). Mines Br., Dep. Energy, Mines Resour.: Ottawa, Can. Successive electrochem. redns. of a vitrain from low-volatile bituminous coal in ethylenediamine satd. with LiCl resulted in the addn. of 53 H atoms per 100 C atoms. H added at the beginning had a much greater effect on soly. than H added later. The removal of S took place only after the more reactive aromatic rings were reduced. C. McGehee.

1966 129768e Desulfurization of coal. Lee, Bernard S. S.; Schora, Frank C., Jr. (Institute of Gas Technology) U.S. 3,640,016 (Cl. 44/1; C 10 1), 08 Feb 1972, Appl. 579,923, 16 Sept 1966; 5 pp. When a mixt. contg. an equal vol. of a S-bearing coal and calcined limestone of 16-80 mesh size is treated with H in a fluidized bed at 900-800° and atm. pressure, substantially all pyritic-S, ~34% of the org. S, and some sulfate

S are removed. The caking bituminous coal originally contained the following wt. % S: pyritic 1.92, org. 1.78, and sulfate 0.18.

1965

8.25 37662t Desulfurizing carbonaceous materials. Murphy, Richard M.; Messman, Henry C. (Carbon Co.) U. S. 3,387,941 (Cl. 23-209.9), 11 Jun 1968, Appl. 23 Mar 1965; 3 pp. In materials like coal, char, coke, or petroleum, the S content is 35-50% decreased by treatment with an alkali metal hydroxide, oxide, carbide, carbonate, or hydride at 500-850°, i.e. above the m.p. of the hydroxide of the alkali metal used. A fluidized-bed system is used. The S-contg. impurities are volatilized during the treatment. Intermittent introduction of steam aids in removal of most of the S-compds.; H₂O washing removes most of the remaining S. Thus, a heavy (no. 6 or Bunker C-grade) petroleum fuel oil with 3.2% S, was heated to 350°, then slowly injected into molten NaOH at 500-25°, causing 75% of the oil to vaporize. The heavy oil floating on NaOH was decanted, cooled, and washed with H₂O. It then contained 1.5% S. The vaporized oil, after condensing and washing with NaOH and H₂O, contained 0.9% S.

8.26 Hydrotreating coal oils to fuels for supersonic aircraft. C. O. Hawk, M. D. Schlesinger, Paul Dobransky, and R. W. Hiteshue (U. S. Bur. of Mines, Pittsburgh, Pa.). U.S. Bur. Mines, Rept. Invest. No. 6655(8), 31 pp. (1965) (Eng). At speeds of Mach 3 it is estd. that airframe temps. will reach 300-500°. Suitability of fuel to serve as a heatsink while retaining good combustion properties depends on the sp. heat and thermal stability from 150 to 300° or above. Important also are high heats of combustion and high ds. (for space-limited craft), high b.ps. (for low vapor pressures at the high temps. likely to exist in flight), low f.ps., and low viscosities at low temps. Distillable oils derived from coals were desulfurized and hydrogenated at 2500 psig. in a lab. 2-step vapor-phase catalytic operation to give a product rich in satd. cyclic hydrocarbons. The 1st step was mainly desulfurization at 400°, the 2nd satn. at 300°. One of the oils used as feedstock was from low-temp. carbonization of a bituminous coal, the other was from liquid-phase hydrogenation of coal in a pilot plant. The product from the satn. step was distd. to remove the light ends and high-boiling residue and to recover a fraction having acceptable properties with respect to heating value, d., f.p., viscosity, and boiling range according to Air Force specifications.

8.27 Catalytic hydrogenation of low-temperature tar fractions. Mrs. A. Mirza, A. V. Ramaswamy, M. Aziz Masood, S. A. Qader, and R. Vaidyeswaran (Regional Res. Lab., Hyderabad).

Indian J. Technol. 3(2), 57-60 (1965) (Eng). Cylindrical pellets of catalysts contg. oxides of Co and Mo and sulfides of W and Ni supported on Al_2O_3 were compared on the basis of their hydrogenation characteristics. One main advantage of these catalysts is their resistance to S and N compds. The fraction of low-temp. tar with 200-350° b.p. formed the feed materials without and after repeated extn. with 10% NaOH and 1:1 dil. HCl. Hydrogenation was done at 350-480°, at 70 and 100 kg./cm.², and at space velocities of 0.5-2.0 vols./vol. catalyst/hr. With both catalysts as the hydrogenation temp. was increased, the contents of tar acids, N, and S decreased and the amt. of the fraction b. <200° increased due to the hydrogenolysis of O, N, and S compds. and the cracking of hydrocarbons, which increased with reaction temp. The acids are more amenable to hydrogenation than S compds., which in turn are more easily hydrogenated than N compds. The 2 catalysts showed little difference in activity in the hydrogenation of tar acids and S compds. At 410° and 100 kg./cm.², tar acids are completely removed and the extent of redn. of S is ~90%. Ni W sulfide is more active and effective at lower temps. than Co molybdate in the hydrogenation of N compds. With both catalysts, there is an increase in the tar-base content in the initial stages and as the temp. is increased, the base content decreases progressively, while the total N content falls throughout. The catalysts did not exhibit any significant activity towards the hydrogenation of aromatic hydrocarbons under the exptl. conditions employed, although they are very effective towards tar acids, N and S compds., and olefins. Ni W sulfide is more active towards aromatics, as shown by the higher aniline points and satd. hydrocarbon content of the product obtained. High temps. (450-80°) are necessary for the complete removal of O, N, and S compds. with both catalysts. In any case, the product obtained either from the tar fraction or neutral oil is not suitable for use in high-speed diesel engines or as an illuminant. The refined product from the tar fraction requires secondary processing due to the higher aromatic content; hence neutral oil should be a better raw material for the production of middle distillates. Ni W sulfide appears to be more suitable for the hydrogenation of low-temp. tars.

1961

8.28 Synthetic fuel from coal for supersonic aircraft. M. D. Schlesinger and R. W. Hiteshue (U.S. Bur. of Mines, Bruceton, Pa.). U.S. Bur. Mines, Rept. Invest. No. 5902, 19 pp. (1961).--The tar oil used was a 230° to 300°C. fraction of low-temp. tar obtained by carbonization of a West Virginia bituminous coal in the pilot plant of the Consolidation Coal Co. The desulfurization at 400°C. and satn. at 300°C. were

done in the vapor phase at 2500 p.s.i.g. After catalytic treatment and distn. the final jet fuel had a b. range of 194° to 247°C., a net heating value of 18,420 B.T.U./lb. and a sp. gr. of 0.8463 60°F./60°F. It also had a pour point below -62°C., and at 0°C. its viscosity was 4.5 cp. The oil was virtually all bicyclic, 41% noncondensed and 56% condensed. Recovery of the final product amounted to 23% of the feed to the desulfurization step.