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CaC₂ Production from CaO and Coal or Hydrocarbons in a Rotating-Arc Reactor

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Calcium carbide was produced by passing premixed suspensions of finely divided lime in methane or ethylene, or of finely divided lime and a bituminous coal in hydrogen, through a rotating-arc reactor. Calcium carbide is thermodynamically stable from arc temperatures down to room temperature and thus can be recovered for subsequent conversion to acetylene by reaction with water. It is postulated that active species such as free radicals formed in the arc from the hydrocarbon gases or the coal react with lime to form carbide. The fractional lime conversion to carbide is determined by the power into the arc, and a threshold power exists below with no carbide forms. A limiting lime conversion of about 70% is found, possibly reflecting the buildup of a carbide layer that is essentially impervious to the reactive hydrocarbon species in the short time available in the arc. Under appropriate conditions of power and lime/hydrocarbon or coal feed ratio, carbon conversion to carbide is 100% for methane and ethylene and over 90% for the coal.

Introduction

In recent years, gasification of finely divided coal, especially in hydrogen atmospheres, has been studied extensively. The fraction volatilized has been found to increase with increased partial pressures of hydrogen and increased final temperatures. Fine grinding and extensive dispersion of the coal in the gas phase also benefit volatiles yields by reducing hydrocarbon cracking and the associated formation of char and soot. Acetylene content of the volatiles increases with temperature until it finally becomes the predominant component. Thus Nicholson and Littleweed (1972) explored the gasification of a high volatile coal in argon containing 10% hydrogen by passing coal-gas suspensions directly through a dc arc at atmospheric pressure. Up to 94% of the coal's carbon was volatilized, with 74% of this carbon appearing as acetylene. Bond et al. (1966), Graves et al. (1966), and Kawana et al. (1967) reported similar results though with somewhat smaller conversions. Krukonis (1968) and Gannon and Krukonis (1972) produced acetylene by passing a suspension of finely divided high volatile coal in hydrogen through a magnetically rotated arc and discussed the economic feasibility of such a scheme for industrial operation. Since yields with anthracite were far smaller than with bituminous coal, acetylene appears to form from the coal volatiles produced during rapid heating in the arc, rather than from direct reaction of solid carbon with hydrogen. All these studies indicate that, at the high temperatures of an arc, vaporized hydrocarbons crack to form free radicals, etc., which then participate in gas-solid reactions. This was further shown by the work of Stokes (1971), who passed a suspension of

fine particles of Ta₂O₅ in methane into a helium plasma jet, and attained up to 42% conversion to Ta, TaC, and Ta₂C. Similar findings were made with dispersions of WO₃

From a consideration of these results, it appeared possible that calcium carbide might be made in an arc by injecting a dispersion of CaO in methane, or by a dispersion of CaO and coal in a suitable gas. Unlike C₂H₂, calcium carbide is thermodynamically stable from arc temperatures down to room temperature; hence the chances of preserving any carbide produced seemed reasonable. The following overall reactions are involved in carbide formation, although they do not, of course, represent the actual pathways followed.

$$CaO + 3C = CaC_2 + CO$$
 (1)

$$CaO + 3CH_4 = CaC_2 + CO + 6H_2$$
 (2)

In reaction 1, ΔH at 2000 °C = +106.5 kcal and $K \geq 1$ at >1900 °C. In reaction 2, ΔH at 2000 °C = +172.8 kcal and $K \ge 1$ at >1200 °C. Inspection shows that these reactions are endothermal (i.e., heat of reaction $\Delta H > 0$), and develop favorable equilibrium constants (K) only at elevated temperatures. This study was therefore undertaken to explore the possibility of producing carbide in gas solid suspensions in a high temperature arc (Kim, 1977).

In this entrained flow reaction system, the formation of carbide from lime and coal is presumably preceded by the formation of hydrocarbon volatiles from coal, which may then react with lime to form carbide. Therefore, to aid in understanding of the reaction, methane was extensively studied as a carbon source despite its unusually high

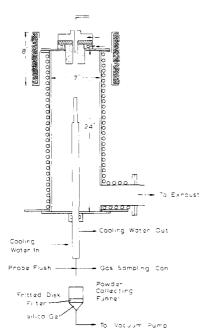


Figure 1. Simplified overall view of the reactor.

energy requirement due to its negative heat of formation and the high H/C ratio.

Experimental Section

The general approach involved passing one of the following suspensions of finely divided solids through an arc: CaO in methane, CaO in ethylene, and CaO plus coal in hydrogen. The solid products were recovered and analyzed for CaC2. The CaO was of two types and of three particle sizes: reagent grade (-8 µm) and a calcine of a high-purity natural calcite (-44 and -74 μ m). The coal was a Pittsburgh Seam No. 8 high volatile bituminous (38.9% volatiles, 1.4% moisture, 11.3% ash, as-received) analyzing 67.8% C and 4.8% H, dry ash-free. The methane, ethylene, and hydrogen used as feed gases were of reagent grade.

Reactor. A schematic view of the arc reactor is presented in Figure 1, and of the head assembly in Figure 2. The solenoid concentrically surrounding the reactor provides a magnetic flux of about 120 G to rotate the arc, which itself is operated at power levels up to 27 kW. By being rotated, the arc is stabilized, overheating of parts of the electrodes is reduced, and gas by-passing is diminished.

As shown in Figure 2, the cathode is a 1/4-in. graphite rod, positioned in the center of the anode insert. This insert is a hollow graphite cylinder, 1/2 in. i.d., 1 in. o.d. and 1 in. long, and is held in position by the so-called anode "support tube." The arc forms in the space immediately below the end of the cathode, as sketched, extends only a short way down into the anode insert, and lengthens a trifle with gas velocity. The gas-solid suspension leaving the arc remains at high temperature while it travels down through the support tube below the anode insert and then cools suddenly as it exits into the chamber below, which is surrounded by cooling coils.

Residence times of the gas-solid product stream leaving the arc are varied by using support tubes of different lengths. Residence times in the cooling chamber are independently varied by raising and lowering the ¹/₄ in. o.d. copper water-jacketed sampling tube, in which the sample withdrawn is rapidly cooled.

The possibility of nonuniform radial distribution of the solid product in the cooling chamber was examined by

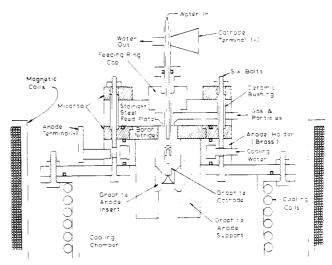


Figure 2. Schematic of rotating-arc reactor head assembly.

analyzing for CaC₂ the particles collected from the bottom and the sidewalls of the chamber and comparing the results with those from the sampling funnel collected at a 15 in. quench distance. The examination revealed that the percentage conversion of CaO to CaC2 was lower by typically 2-3% and by as much as 7% for the samples collected from the bottom and the side walls in comparison with those from the sampling funnel. This discrepancy is attributed to the hydrolysis of the CaC₂ product inside the cooling chamber by the moisture in the open air, which was let in during an approximately 3 min period required to dismantle the reactor and to collect the powders from the inside.

Solids Feeder. The solids feeding device is a gas-tight box containing a mechanically vibrated hopper which unloads powder onto a conveyer belt. This belt discharges into a chute, where the powders are picked up by the gas and carried into the reactor opening shown in Figure 2. This powder suspension, initially at room temperature and pressure, passes downward through the arc and out into the cooling chamber below.

Operating Procedure. The reactor is first brought to temperature by initiating the arc in argon and then slowly switching over to the gas of interest (e.g., CH_4 , C_2H_4 , or H_2) after which the solid feed is started. The feed rates of both the gas and solid feeds are adjusted to the desired levels prior to reactor operation. Each run lasts from 1 to 3 min, which is easily adequate for a reaction which is apparently completed in the 10^{-5} to 10^{-2} s required by the gas to flow through the arc and support tube.

The power supplied to the arc at any time equals the product of the arc voltage and arc current. Since these fluctuate widely and rapidly, the averages of each are used to determine the power input in kilowatts. This calculated power in kilowatts is, of course, numerically identical with the energy supplied per hour, expressed as kilowatt hours per hour. The voltage-current behavior of the arc when feeding a suspension of CaO in methane, as shown in Figure 3, is typically that of a low to medium intensity arc. Inspection of this figure shows that doubling the gas flow rate from 16-17.3 L/min to 30-35.8 L/min increases the voltage for a given current flow. This increased voltage is presumably necessary to supply the energy needs of the greater gas flow, and possibly also to overcome any increased resistance in the slightly lengthened arc. The arc is most stable when gas flow rates are low and power inputs exceed 16 kW or so, under which conditions the power fluctuates about 3-10% around the mean. These fluctuations increase as gas flow is increased at constant power

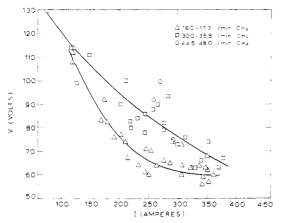


Figure 3. Voltage-current characteristics of the rotating arc in methane with lime feeding.

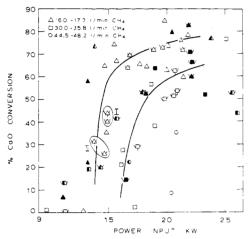


Figure 4. Effect of power input on lime conversion to carbide at different methane flow rates [lime particle diameter: $-8 \mu m (\Delta, \Box, O)$; $-44 \mu m (\Delta, \blacksquare, \bullet)$; $-74 \mu m (\Delta, \blacksquare, \bullet)$; quench distance: 5 in. (points without arms); 15 in. (points with arms); residence time: regular (points without tails); extended (points with tails)].

and rise to about 30%, beyond which level the arc can be sustained only with extreme difficulty.

The solid sample collected by the probe is thoroughly mixed, after which the CaC₂ content is determined by chromatographically measuring the acetylene evolved upon water addition. The total calcium content of the product is determined by the method of Snell and Ettre (1969). Analytical results are found to be highly reproducible.

The product gas is analyzed for hydrocarbons from time to time by chromatography. As further discussed later for methane runs, when using lime in about 30% excess over stoichiometric for CaC_2 production from the methane fed with sufficient power input, then the off-gases are found to contain no hydrocarbons presumably leaving H_2 and CO as the only gas products, with carbon conversion to CaC_2 (and CO) close to 100%. At smaller lime-methane ratios, C_2H_2 and other light carbons are found in small concentrations in the off-gases, together with H_2 and CO. In the coal runs only CO and CO are found in the off-gases.

Methane–Lime Runs. In the initial runs, suspensions of lime in methane were passed through the arc, whereupon finely divided, highly hygroscopic, and easily oxidized calcium carbide was found in the solid product along with unconverted lime and solid carbon. The effect of increased power input on CaO conversion to carbide for three different methane feed rates of 16–17.3, 30–35.8, and 44.5–48.2 L/min is presented in Figure 4. Inspection shows that at all lime feed rates from 0.04 to 0.40 mol/mol of CH₄, the fractional lime conversion to carbide is de-

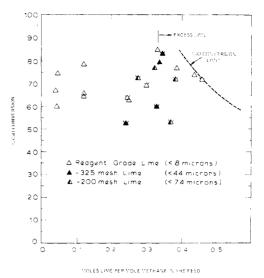


Figure 5. Conversion of lime to carbide in methane at different lime/methane feed ratios [power input, 15.5 to 23.8 kW; methane flow rate, 16.0 to 17.3 L/min; quench distance: 5 in. (points without arms); 15 in. (points with arms)].

termined solely by the power into the arc. Since carbide formation is by reaction 2, stoichiometric lime feed would be 0.33 mol/mol of methane. By further inspection, it appears that for each constant methane feed rate, a threshold power exists below which no carbide forms. Above this power threshold, lime conversion increases rapidly to about $70\% \pm 15\%$ for a small further increase in power, after which conversion remains constant at about 70%, regardless of the power input. Thus at a methane flow of 16-17.3 L/min, as shown by the data points of Figure 4, this critical power range is from 14 to 16 kW at all lime feed rates, while at 30-35.8 L of methane/min, this critical range is about 16-19 kW.

For the constant methane flow of 16–17.3 L/min, at all power levels above the critical, lime conversion is shown plotted against lime feed rate in Figure 5. The region of excess lime, where the lime/methane feed ratio exceeds the stoichiometric value of 0.33 according to reaction 2, is indicated in the figure. Within this region, the theoretical lime conversion limit corresponding to complete conversion of methane carbon to carbide carbon is given by the dashed curve. Inspection shows that the observed lime conversion of $70\% \pm 15\%$ is independent of the lime feed rate as well as of the power level. This generalization also applies to runs made at higher methane flows.

In passage through the arc, methane presumably decomposes according to the following overall reaction sequence (Kahn and Crynes, 1970; Miller, 1965, pp 371–378)

The initiation step of the methane decomposition is generally believed to involve either or both of the following reactions (Chen et al., 1975; Hartig et al., 1971)

$$CH_4 \rightarrow CH_3 + H$$

 $CH_4 \rightarrow CH_2 + H_2$

It is the reactive intermediates and nascent carbons formed in these reactions which presumably react with the lime to form calcium carbide. When lime conversion is constant at about 70%, then the amount of carbide formed at a given total methane feed, and therefore the amount of methane reacting to form carbide, is directly propor-

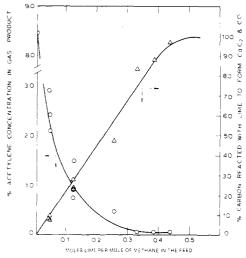


Figure 6. Effect of lime feed rate on acetylene concentration in product gas and extent of carbon conversion in methane runs (methane flow rate, 16.0–17.3 L/min; power input, 15.5–21.8 kW; quench probe position, 5 in.; reagent grade lime).

tional to the lime feed rate. This proportionality of methane reacted (to carbide) and lime feed rate for all power levels above the critical is shown by the data points of Figure 6 for a constant methane flow of 16–17.3 L/min. It is obvious that conversion of the feed methane to carbon is substantially complete when feeding 0.45 mol of lime/mol of methane—this follows directly from the fact that lime conversion is constant at about 70% and that carbide formation is by reaction 2. Note that no runs were made with a lime/methane feed in excess of 0.45.

Acetylene concentrations in the off-gas vs. lime feed for a methane feed rate of 16-17.3 L/min are shown also in Figure 6. When no lime is fed, the off-gas analysis is found to vary somewhat with the position of the sampling tube. When the sampling point is typically 5 in. below the anode support tube, the off-gas is found to contain 8.5% C₂H₂ (see Figure 6), 1.2% C_2H_4 , 0.4 CH_4 , and the rest H_2 . In the presence of even a little lime, no methane is found to survive the arc for power levels above the critical. The acetylene (and ethylene) concentrations fall rapidly toward zero as the lime-methane feed ratio is increased so that upon attaining a molal feed ratio for lime/CH₄ of 0.4 the solid product becomes a mixture of CaC₂ and CaO, while the off-gas is free of hydrocarbons and presumably contain H₂ and CO only. Under these feed conditions, substantially all the carbon in the methane feed and 70% of the lime is converted to calcium carbide.

Further inspection of the data points of Figures 4 and 5 shows that conversion of lime to carbide was little affected by lime particle size, with the $-74~\mu m$ particles perhaps undergoing slightly lower conversion than the two smaller sizes. Again, neither prolonged "high temperature residence time" in longer anodes, or in the cooling chamber, had any noticeable effect on conversion.

Ethylene-Lime Runs. A few ethylene-lime runs were made, involving gas feed rates of either 15.8 or 21.9 L/min, with lime feed rates of 0.95 to 1.2 times stoichiometric. Results, as discussed later, are similar to those of the methane runs.

Coals Runs. The feed here was always a mixture of -170 + 325 mesh (44 to 88 μ m) coal with either reagent grade lime of -8 μ m particle size, or calcined limestone of -44 μ m size. In most runs, the feed ratio of atoms carbon in the coal per mole of CaO was 2.0 (theoretically, CaC₂ production by reaction 1 requires 3 atoms carbon per mole of CaO), but in a few runs this ratio was 3, and in another

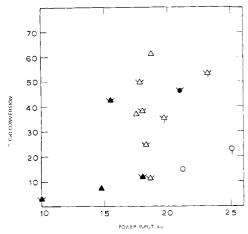


Figure 7. Lime conversion to carbide in runs with coal at different power inputs and lime/carbon feed ratios [excess lime, molar CaO/C = 1.47/3.00 (\triangle , \triangle); excess carbon, molar CaO/C = 1.00/3.06 (\bigcirc , \bigcirc); anode: graphite (\triangle , \bigcirc); copper (\triangle , \bigcirc); quench distance: 5 in. (points without arms); 15 in. (points with arms); residence time: regular (points without tails); extended (points with tails)].

it was unity. The mixed-powder feed rate ranged from 15.5 to 51 g/min, and the hydrogen flow rate from 14.6 to 48.6 L/min.

Results of the 15 coal runs made are plotted in Figure 7 and again show CaC_2 to have been produced. A serious scatter of results was encountered, in that repeat runs gave significantly different lime conversions. This nonreproducibility was probably the result of nonuniform solid feed resulting from coal sticking to the hot graphite walls of the anode insert holder. This sticking could be avoided by using a hollow copper anode holder through which cooling water circulated, but frequent failure of this system by burn-out discouraged its use. Significantly, however, in the most successful runs, 91% conversion of the coal's carbon to calcium carbide was attained when feeding 20.1 g/min of mixed powder (containing 2 atoms carbon per mole of CaO) and 28 L of H_2/min .

Discussion

Methane Runs. In all runs, regardless of the initial particle size or type of lime, solids emerged from the arc reduced to the same size range of less than $0.3~\mu m$. Such diminution to the same size range occurred, regardless of the fractional conversion of lime to CaC_2 , as illustrated by Figure 8. The cause of this size reduction of the lime particles is not clear, but may be thermal shock due to rapid heating in the arc. Coal particles in the coal runs also appeared to show some size reduction. Soot particles found in the recovered solids were formed by hydrocarbon cracking, and were always of order $0.03~\mu m$ in size.

A possible explanation can now be proposed for the findings that percent conversion of lime to carbide at higher power rates is constant at $70\% \pm 15\%$, regardless of initial lime particle size, of lime feed rates, and of residence times downstream from the arc. It is postulated that reactive species such as free radicals form in the arc from the hydrocarbon gases or the coal feed to the system. These reactive species disappear either by cracking or by reacting with lime to form carbide. Carbide formation is presumed to be the faster reaction, but it comes to an end when a lime particle becomes surrounded by an impervious layer of CaC₂. Whether the lime particles themselves or grains within these particles are effectively the reacting entities depends on the rate-controlling mechanism, which is not established. Nevertheless, the absence of a significant particle size effect can be rationalized in either case, as follows. If the overall rate is jointly controlled by

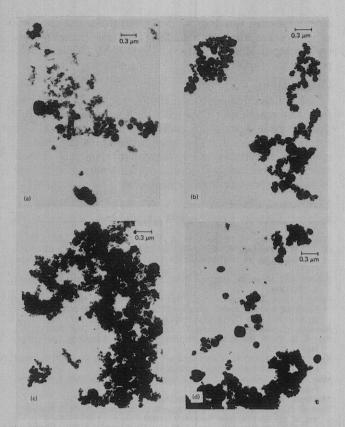


Figure 8. Solid material from arc after different fractional conversions of lime to carbide [lime conversion: (a) 7.0%; (b) 21.7%; (c) 58.3%; (d) 79.5%; feed particle size, -325 mesh (44 μ m); methane feed rate, 16.7 L/min; lime feed, stoichiometric; power input: (a) 11.1 kW; (b) 13.2 kW; (c) 13.3 kW; (d) 20.5 kW].

diffusion and reaction in the pores of the particle, the particle itself would develop the CaC2 layer. Since all lime particles break up into the same size range in the arc, regardless of feed sizes, the rate would not depend on particle size. Alternatively, if pore diffusion is fast enough to allow uniform exposure of the surface of all grains throughout the particle to the gaseous reactants, the buildup of CaC₂ would occur on the grains. Particle size would again not influence the rate since any mass transport limitation would then lie within the grains, the size of which seems to be independent of particle size. Regardless of which type of layering actually occurs, a constant conversion (70% \pm 15% in this case) is the result when the power levels were above the critical. In consequence, prolonged residence time of the reacting system downstream from the arc has no further effect on percent conversion of lime to carbide.

In addition, reactive species such as free radical which are presumably responsible for the CaC₂ formation have short lifetimes and may not exist very far downstream of the arc. This condition could also lead to no effect of prolonged residence time on the conversion of CaO to CaC₂. Support for this suggestion is provided by the data, marked by two circles designated I and II in Figure 4, from two sets of experiments at the critical power levels in which the residence time was varied by a factor of 13 by using anode support tubes of different length. Although the CaO conversions are sufficiently lower than the plateau value of 70% to suggest that an imperviously thick layer of CaC₂ might not have developed, the prolonged residence time apparently led to no further CaO conversion. calcium carbide production is determined by the lime feed rather than the carbon (hydrocarbon) feed. Since onethird of a mole of CaO is theoretically needed per mole of

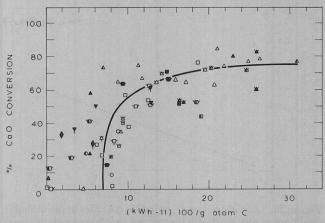


Figure 9. Lime conversion to carbide in methane and ethylene under different conditions as correlated with the reduced specific energy input [gas feed rate: methane runs, 16.0-17.3 L/min (Δ) 30.0-35.8 $L/\min(\Box)$, 44.5–48.3 L/min (O); ethylene runs, 15.8 L/min (∇), 21.9 L/min (\spadesuit); lime particle diameter: $-8 \mu m$ (\triangle , \square , O); $-44 \mu m$ (\blacktriangle , \blacksquare , •); $-74 \ \mu m$ (\triangle , \square , \bullet); quench distance: 5 in. (points without arms); 15 in. (points with arms); residence time: regular (points without tails); extended (points with tails)].

carbon to form a mole of CaC2 by reaction 1, then with 70% lime utilization, complete carbon utilization can be attained by feeding 0.33/0.7 or 0.48 mol of CaO per mole of carbon in the gas. This performance was approximated in several runs.

Further perspective on the methane and ethylene runs has been developed by replotting the data points of Figure 4 and shown in Figure 9. On this latter graph, percent CaO conversion to carbide is plotted against X, where X is defined as

$$X = \frac{(\text{kWh} - 11) \times 100}{\text{gram-atoms of C in feed}}$$

By this correlation, despite the considerable scatter of the data, the points of all the methane and ethylene runs fall quite well within a band around a single line. The constant (11) in this expression for X was found by curve fitting procedures. The possible significance of this constant can only be guessed at, but it might represent not only apparatus energy losses but also that energy which must be exceeded before generating those particularly reactive species needed to convert CaO to CaC2.

Inspection of Figure 9 shows an abrupt improvement in lime conversion at an X value of about 8. The existence of such a threshold power input can be interpreted in several ways. It may be that a minimum arc temperature is needed to develop a sufficiently porous CaC2 shell around the lime particles to allow the penetration of the reactive species needed for the 70% conversion already discussed. Alternatively, it may be needed to generate an especially reactive gas species needed for kinetic reasons to produce carbide.

Ethylene Runs. The ethylene-lime runs are also plotted in Figure 9. Examination shows that these points fall more or less into the region of the methane data, indicating that the same general conclusions apply.

Coal Runs. The coal-hydrogen data are replotted as percent lime conversion vs. X in Figure 10. Inspection shows that the data now fall more nearly around a single line, again suggesting that the constant 11 has significance much as in the methane and ethylene runs. There is little evidence, however, of a critical threshold power level such as was observed in the methane runs. Moreover, the methane runs are all with a deficit of lime relative to the carbon fed (mol of lime/atom C of less than 3), while the

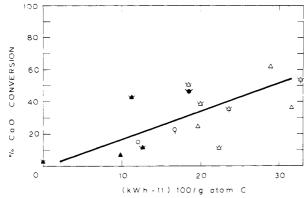


Figure 10. Lime conversion to carbide in coal runs under different conditions as correlated with the reduced specific energy input [feed mixture: excess lime (Δ, Δ) ; excess carbon (O, \bullet) ; anode: graphite (Δ, O) ; copper (Δ, \bullet) ; quench distance: 5 in. (points without arms); 15 in. (points with arms); residence time: regular (points without tails; extended (points with tails)].

coal runs are all with an excess of lime (mol of lime/atom C of more than 3); hence comparisons of these runs are difficult. The energy requirements in the best run, equivalent to 36 kWh/lb of acetylene producible from the carbide formed, would presumably be improved in larger scale apparatus. In the present method the theoretical energy requirement would be 4.0 kWhr/lb of C2H2 with coal as a carbon source if the reactants were fed at room temperature and the feed C/H ratio was 1/2.

Extremely short reaction time and the possibility of producing CaC₂ directly from coal or hydrocarbons without having first to produce coke appear to make the present method more advantageous than the conventional method of manufacturing CaC2 from lime and coke in large

electrothermal furnaces if energy requirements for both processes are about the same. The latter requires an energy input of about 5.0 kWh/lb of C₂H₂ (Miller, 1965, p 181).

Conclusions

It has been demonstrated that CaC2 can be produced from the reaction of CaO with high volatile coal or hydrocarbons in a one-stage reaction by using a magnetically rotated arc reactor. The results illustrate the intense chemical activity of the nascent hydrogen and carbon species concomitantly produced during the intense heating of hydrocarbons or high volatile coal.

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Pilot Plant Results for Partial Oxidation of Cattle Feedlot Manure

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A small pilot plant (capacity, 450 kg/day) has been constructed to evaluate gasification of manure by partial oxidation. The reactor operates as a countercurrent, fluidized bed in which an air-steam mixture is the fluidizing medium and manure is the only solid phase present. The product gas from this process contains a significant amount of hydrocarbons (CH₄ \sim 10 %, C₂H₄ \sim 6%) in addition to normal partial oxidation products. The net heating value of the dry gas exceeded 8.6 MJ/m3 (1 atm, 0 °C) in all cases. The net heating value of the CO2-free gas (still containing nitrogen) exceeded 11.2 MJ/m³. The pilot plant design and operating procedure are described as are the results of the heat and material balances. This work confirms the technical feasibility and potential scale-up for producing a medium heating value gas suitable for fuel or for feed to a petrochemical production process at relatively high yields from cattle feedlot manure.

Introduction

In view of the approaching energy shortage in the United States, new sources of energy must be developed to maintain the standard of living as current energy resources are depleted. Biomass is one of the many possible alternative energy resources. Biomass can be defined as solar energy stored in the form of plant matter or residues which have some positive value as a chemical resource. This is in contrast to wastes which have a negative value; i.e., one must pay to dispose of wastes. Many obstacles stand in the way of development of biomass as an energy source. One prime obstacle is the fact that the energy density of biomass is very low compared to fossil fuels. This results in high collection costs to deliver the biomass to a conversion facility. It is projected (Inman, 1977) that wood can be produced by intensive silvaculture at the rate of