FINAL TECHNICAL REPORT

September 1, 1987 through August 31, 1989

PROJECT TITLE: EXPLOSIVE FRACTURE COMMINUTION PROCESS

FOR COAL CLEANING USING SUPERCRITICAL CO2

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ABSTRACT

Current comminution techniques do not comminute selectively. They do not selectively separate or scission the carbonaceous matter from the mineral matter. Furthermore, only a few percent of the applied energy is utilized for comminution, the rest being dissipated in process inefficiencies. In this project, supercritical CO₂ is being harnessed to explosively fracture the coal into discrete minuscule particles and release the mineral matter without substantially altering the mineral matter's size. The process takes advantage of the porous fluid-permeable nature of the carbonaceous component of the coal. When the coal is flooded with supercritical CO₂, the dense fluid permeates into the pores of the carbonaceous matter in coal without affecting the mineral component. On instantaneous depressurization, the supercritical CO₂ undergoes a sudden phase change and volume expansion causing the carbonaceous matter to literally explode into discrete minuscule particles without altering or reducing the size of the mineral matter.

In year one of the study, explatory work was done on one of the Illinois No. 6 coal (IBCSP sample No. 1). Thus, chunks of IBCSP sample 1 with average particle diameters in the range of 7 to 8.2 mm (7000-8200 microns) was explosively shattered in a micro reactor with supercritical CO₂ using a soak time of 5 minutes and gave minute discrete particles of varying sizes. A comparison of the explosively fractured coal with a mechanically crushed sample (-100 mesh) of the same coal in a float / sink operation of Sp. Gr. 1.3 showed that the ash was evenly distributed in the sink and float fractions (11% and 8% respectively) for the mechanically crushed samples; whereas for the CO₂ fractured sample there was very little ash in the float (2%) and all the ash concentrated in the sink fraction. This suggests selective comminution of the carbonaceous fraction of the coal.

A set of of thirty explosive shattering experiments were performed by varying temperatures and pressures and the fractured coal particles were separated into 13 different fractions by sieving. A graphical analysis showed that at all the three operating temperatures (20, 30, 40 °C), an increase in pressure resulted in the formation of more fractured material in all the sieved fractions. Ash analysis of the sieved fractions showed that the larger particle size fractions (700-4000 microns) contained as much as 50% of the ash.

In year two of the study a newly designed, and larger explosive fracture, unit was fabricated. A reduction in mean mass size of the fractured coal particles from 860 microns to 420 microns on going from 900 psia (subcritical) to 1300 psia (supercritical) at 40°C with 7-8.2 mm particle size feed vividly demonstrated the need to be operating under supercritical conditions. Comparision of the mineral matter particle size before and after explosive shattering showed no significant differences. This showed that only the carbonaceous component of coal was exposively shattered into discrete particles as postulated by us. In the supercritical region, temperature changes do not affect the degree of comminution. However, increasing pressure tends to produce more fine coal. The degree of comminution of IBCSP -2 & 6 is better than the other IBCSP samples.

EXECUTIVE SUMMARY

Physical coal cleaning is achieved by grinding the coal to liberate impurities that are not chemically bound and then taking advantage of specific gravity differences or surface wetting properties between the coal macerals and denser mineral impurities. It is, however, recognized that in the field of crushing and grinding only a few percent of the energy applied to the system is actually used in fracturing the coal; the remainder being dissipated in process inefficiencies., Current techniques do not comminute selectively. They grind both the ash-forming minerals, as well as the carbon fraction of the coal. They also do not selectively separate or scission the carbonaceous matter within the coal from the mineral constituents of the coal. The current comminution techniques are also limited in the degree of size reduction. In fact, for some Illinois coals, grinding is needed to minus 400 mesh (37 microns) for liberation of mineral matter.

In this project, it is proposed to harness carbon dioxide under supercritical conditions to explosively fracture the coal into discrete minuscule particles and release the mineral matter without substantially altering the mineral matter's size. The process takes advantage of the fact that the carbonaceous fraction of coal is a porous fluid permeable solid whereas the mineral component of the coals is nonporous. When the coal is flooded with supercritical CO2, the dense fluid permeates only into the pores of the carbonaceous matter in coal. When an instantaneous depressurization is done, the supercritical CO2 undergoes a phase change with a sudden expansion causing the carbonaceous fraction of coal to literally explode into discrete minuscule particles without altering or reducing the size of the mineral matter of the coal. CO2 is widely distributed in power plant stack gas, as well as in gasification plants. New technology is now available to recover CO2 from flue gas streams.

In related work, supercritical water has been used to fracture the coal for comminution (12). Very high operating temperatures and pressures are used ($T_C = 374^{\circ}C$ and $P_C = 220$ Atms.), and the depressurization has, to be done at 93-149°C. Dissolution and degradation of coal components takes place at this height, temperature, and pressure.In addition, the ISGS while studying the

aggregate flotation (AF) process coupled to the IITRI, explosive shattering (ES) process, report that "volatiles" produced during absorption onto the comminuted particle after shattering and causing them to aggregate. Very little beneficiation was possible with AF because the "as received" ES product tends to clump into 200-500 micron aggregates cancelling the effect of the extensive initial comminution. The CO₂ process operates near room temperatures and so these problems will not arise. Therefore, our fracturing process could potentially be coupled to the ISGS - AF process for coal cleaning.

In the first year of the project chunks of Illinois No.6 coal (IBCSP No. 1) with particles having average diameters in the range of 7 to 8.2 mm was explosively shattered in a micro reactor with supercritical CO2 using a soak time of 5 minutes and gave minute discrete particles of varying sizes. Runs at 1300 psia and 40°C yielded discrete fractured coal particles, the majority of which were in More material was present in the smaller particle the 38-605 micron range. size range i.e. 38-250 microns at 40°C than at 20°C. Ash analysis of the sieved fractions showed that the larger particle size fractions (700-4000 microns) contained as much as 50% of the ash. In the second year of the project, a newly designed and larger, explosive fracture unit was fabricated. For the volume of our reactor as much as 15g of the coal can be explosively shattered with no change in the particle size distribution of the explosively shattered particles. However, if larger amounts of coal were packed into the reactor a greater percentage of larger size coal particles were formed. 100g of coal was explosively shattered and sieved into thirteen different fractions. As shown in Figure 1, a reduction in mean mass size of the fractured coal particles from 860 microns to 420 microns on going from 900 psia (subcritical) to 1300 psia (supercritical) at 40°C with 7-8.2 mm particle size feed.vividly demonstrated the need to be operating under supercritical conditions. In the subcritical region, fracture was favoured at lower temperatures, while the higher (40°C) temperature was most effective in the supercritical region. Comparision of the mineral matter particle size before and after explosive shattering showed no significant differences. This showed that only the carbonaceous component of coal was exposively shattered into discrete particles as postulated by us.

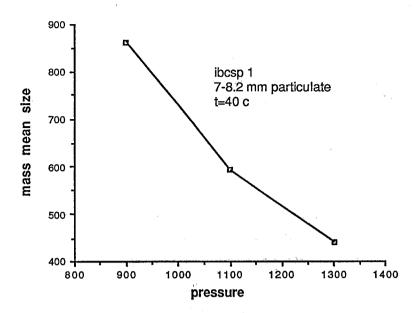


Figure 1. Effect of pressure on mean particle size at 40°C.

Further experimentation showed that the degree of comminution is nearly the same in the temperature range of 29-59°C (with pressure at a constant 1500 psia). However, the runs at subcritical temperatures were not as good for all the IBCSP samples. The increase in pressure has a more dramatic effect on the degree of comminution. On going form 900 psia to 1800 psia more fine coal is produced. Thus, 47.8% of IBCSP coal sample 2 passed through a 80 mesh seive at 1200 psia at 40°C, wheras at 1800 psia 62.1% of coal passed through the 80 mesh seive at the same temperature. The degree of comminution of IBCSP2, IBCSP6 is better than the other other two coal samples. Shattering of moisture free coal gave better comminution results.

The unique potential benefits the CO₂ explosive fracture system has to offer are:

- * No clumping occurs, therefore, separation of carbon from mineral matter is much more effective.
- * Lower process energy inputs.
- * Carbon dioxide readily and extensively penetrates the coal structure. It is likely the CO₂ diffuses into the coal along the lines of mineral inclusion as has been seen for other gases. This is important since the diffusion of

reagents through solid coalmay be the rate-limiting step in many reactions. This diffusion has also been shown to cause a dimensional expansion of the structure.

- * Fracturing and separation can be done in the same CO₂ medium.
- * Can be integerated with the CO₂-coal slurry transportation, gasification and combustion processes and contributing to better process economics of these processes.
- * Attractive to utilities because they produce a lot of CO₂ and can effectively use it for cleaning as well as transporting the coal.
- * The CO₂ functions as a dilutant lowering reaction zone temperatures which reduces NO_x formation

The greatest potential and definitely the most exciting possibility for the project is the coupling of our CO₂ explosive fracture comminution process to the CO₂-coal slurry pipeline concepts. In fact, Southwestern Public Service Co. just finished a successful test run for the operation of a two mile, four feet diameter test loop carrying a coal-CO₂ slurry at its Harrington Station coal-fired power plant near Amarillo, Texas. Thus, the incorporation of a explosive fracture comminution step prior to the transportation would allow more clean coal to be transported.

OBJECTIVES

In this project, it is proposed to harness carbon dioxide under supercritical conditions to explosively fracture the coal into discrete minuscule particles and release the mineral matter without substantially altering the mineral matter's size. This takes advantage of the fact that the carbonaceous fraction of coal is a porous, fluid permeable solid whereas the mineral component of the coal, for example, the pyrites, is nonporous and not permeable by fluids. When the coal is flooded with supercritical CO2, the dense fluid permeates into the pores of the organic matter in coal, the inertia of which is sufficient to prevent the fluid from gradually escaping the pores during the instantaneous depressurization. When an instantaneous depressurization is done, the resulting fluid undergoes a phase change with a sudden expansion, causing the organic component of coal to literally explode. This would result in the formation of discrete minuscule carbonaceous particles without reducing the size of the mineral matter of the coal. The explosive fracture comminution by supercritical CO2 can then be followed by separation of the fractured coal from the intact heavy mineral matter using the dense supercritical carbon dioxide medium itself. Conventional froth flotation processes or cyclone separators can also be employed in the separation step.

BACKGROUND

PHYSICAL COAL CLEANING

Physical coal cleaning is achieved by grinding the coal to liberate impurities that are not chemically bound and then taking advantage of specific gravity differences between the organic matter in coal (the macerals) and denser mineral impurities. It is recognized, however, that in the field of crushing and grinding (comminution):

Only several percent of the energy applied to the system is actually used in fracturing the coal; the remainder is dissipated in process inefficiencies.

Current techniques do not comminute selectively. They grind both the ash forming minerals as well as the carbonaceous fraction of the coal.

This results in the fine mineral matter being intimately mixed and dispersed into the organic phase making separations difficult.

The mechanical or grinding techniques do not selectively separate or scission the carbonaceous matter within the coal from the mineral constituents of the coal.

Current techniques are limited in the degree of size reduction.

CARBON DIOXIDE

CO₂ is widely distributed in power plant stack gas. Hundreds of millions of cubic feet of CO₂ per day is available from this source. The CO₂ content of stack gas varies from 6 to 16 percent depending on the fuel burned. Dow Chemical has announced a two-stage technology that can recovery CO₂ from flue gas streams (1). CO₂ in large amounts can be obtained from gasification plants. About 80 million cubic feet of CO₂ per day are already being produced from two SNG plants in Illinois using naphtha and NGL feedstocks. As much as 300 million cubic feet per day could be generated by some coal gasification plants. This would amount to about a trillion cubic feet over a decade. Integrated combined cycle coal gasification, second generation plants for generation of electricity are also a prime source of CO₂.

The large amounts of CO₂ available, especially as a part of the power plant gases and gasification plants, its unique characteristics, and potential benefits makes the explosive fracture comminution process a very attractive and viable proposition. Thus, the project is directed towards studying *Explosive Fracture Comminution Using Supercritical Carbon Dioxide*.

RELATED TECHNOLOGY

A related project using supercritical water is being developed under DOE sponsorship but this approach represents an advancement over that process and offers much more advantages. Using the supercritical water technology very high operating temperatures and pressures are required ($T_C = 374^{\circ}C$ and $P_C = 220$ Atms.), and the depressurization has to be done at 93-149°C. Dissolution and degradation of coal components takes place at this high temperature, and pressure.In addition, the Illinois State Geological Survey

(ISGS) while studying the aggregate flotation (AF) process coupled to the IITRI, explosive shattering (ES) process, report that *volatiles* produced during the shattering absorb onto the comminuted particle causing them to aggregate. Very little beneficiation was possible with AF because the *as received* ES product tends to clump into 200-500 micron aggregated cancelling the effect of the extensive initial comminution.

SUPERCRITICAL CARBON DIOXIDE TECHNOLOGY

The CO₂ process operates near room temperatures and so the problems discussed earlier will not arise. Therefore, our fracturing process could potentially be coupled to the ISGS AF process for coal cleaning.

The unique potential benefits the CO₂ fracture system has to offer are:

Lower process energy inputs--operating temperature and pressure, $T_{\rm C}$ and $P_{\rm C}$ of ${\rm CO_2}$ are 31.1°C and 72.9 atm, respectively. All the energy is harnessed to rupture the coal particles, unlike in most mechanical energy grinding processes where much of it is dissipated in process inefficiencies.

The CO₂-explosive fracturing process can be integrated with the CO₂-coal slurry pipeline transportation concepts currently being developed. In fact, plans are afoot for construction of a CO₂-coal pipeline to transport Powder River coal to the West Coast for export (2). More recently Southwestern Public Service Co. is operating a two mile 4'-diameter test loop at its Harrington Station coal-fired power plant near Amarillo. The company has been running supercritical CO₂ through the line and even pushing batches of coal through the line (3,4). Thus, the adaptation of the CO₂-explosive fracturing process as a pretreatment step prior to transportation would result in transporting more coal than ash or mineral matter and add to the economics of the coal-CO₂ slurry transportation.

A pumpable coal slurry with high coal loading (80% by weight of coal) can be prepared in CO₂ media which can be fed directly into a combustion/gasification reactor, eliminating the need for dry coal handling with its associated problems. In this connection, Argonne's

EES Division has shown that coal can be burned efficiently in a coal-CO₂-oxygen mixture instead of coal-air mixtures in boilers using small scale (400,000 BTU/hr. experiments) (5). The use of coal-CO₂ slurries for gasification has also been demonstrated (6).

Integrated with other CO₂ related beneficiation technologies like the LICADO process (7), and the Brookhaven Laboratory CO₂-water beneficiation process (8). It will also enhance the capabilities of these processes.

Work reported at the recent University Coal Research Contractors' Conference (9,10) has shown that CO₂-treated coals are better candidates for froth flotation, bubble flotation cleaning techniques to remove ash mineral matter from the coals than non-CO₂-treated coals.

Carbon dioxide readily and extensively penetrates the coal structure (11). In fact, this has led to the advocacy of employing carbon dioxide to measure the internal area of coals. It is likely the CO₂ diffuses into the coal along the lines of mineral inclusion as has been seen for other gases (12). This is important since the diffusion of reagents through solid coal may be the rate-limiting step in many reactions. This diffusion has also been shown to cause a dimensional expansion of the structure.

This process provides a water free coal product.

By using CO_2 -coal slurry systems, the CO_2 can function as a diluant, lowering reaction zone temperatures which in turn **reduces** NO_X formation.

Attractive to utilities because they produce a lot of CO₂ and can effectively use it for cleaning as well as transportation.

EXPERIMENTAL PROCEDURES

RATIONALE

A supercritical fluid (SCF) is a material heated to or beyond its critical temperature (T_C) under a pressure equal to or higher than its critical pressure (Pc). In the supercritical state the fluids exhibit a combination of physical and chemical properties that allow them to become very powerful and unusual solvents. Under supercritical conditions the CO2 would function as a high energy dense fluid (ca. density 0.47 g cm⁻¹. It has higher diffusivity, lower viscosity and lower surface tension than the liquid phase) and would permeate within the pores of the carbonaceous matter in coal, without being able to permeate the mineral matter of coal. The dense fluid mass forms a column of fluid within the pores of the coal, the inertia of which is sufficient to prevent the fluid from gradually escaping the pores during an instantaneous decompression. When instantaneous depressurization is done, the resulting fluid expands rapidly, if not instantaneously, thereby causing the carbonaceous particles to literally explode from within. This would result in the formation of discrete minuscule carbonaceous particles without reducing the size of mineral matter in the coal.

EXPERIMENTAL SETUP

The experimental setup as outlined in our proposal has been modified to incorporate a High Pressure Generator on line to deliver the CO₂ (Figure 1). The high pressure generator is a manually operated piston screw pump designed for compressing the CO₂ to desired pressures before delivering into the reactor. The reactor is a 137 cm³ volume micro-reactor fitted with a thermocouple controlled heating tape. The reactor is encased in a fiberglass chamber which serves as a collector for the fractured coal particles. The key element in the reactor is a trap door held by a cam which comprises the **entire bottom part** of the reactor. Turning the cam past its set point would abruptly release the trap door resulting in a instant depressurization of the reactor. The releasing of the entire bottom part of the reactor and the discharging of the coal into a large collection chamber **ensures** that the coal fracture is a result of explosive forces from within the particles (due to the CO₂ expansion and phase

change) and not because the coal was ejected like a high velocity projectile from a narrow orifice. If the latter event is not prevented, then, both the carbonaceous and the mineral fractions would be fractured. 4 g of coal was charged into the reactor, which was then pressurized with CO2. In the second phase of our work a newly designed and larger explosive fracture unit was fabricated (Figure 2). The reactor could hold upto 100g of coal. As in the earlier reactor the key element was the trap door held by a cam which comprised the entire bottom part of the reactor. The containment chamber which also serves as the collection chamber has a series of baffles at either end of the chamber. The end of the baffles are open to the outside so when the trap door is released instantaneous depressurization to atmospheric pressure occurs. The baffles ensure that all the shattered coal particles are trapped in the collection chamber. Different pressures and temperatures were investigated.

RESULTS AND DISCUSSION

Chunks of Illinois No.6 coal (IBCSP No. 1) with particles having average diameters in the range of 7 to 8.2 mm was selected for the explosive fracture experiments. The particle diameters were determined to serve as a control reference point for the range of coal particles sizes to be used in our experiments. The particle diameters were calculated using the equation ${\rm d}^3{\rm p}=$ average weight / 0.166 pi * density which necessarily assumes that the particles are spheres.

In the first experimental set, the reactor was charged with CO₂ at 1050 psi, the temperature was 31°C and a soak time of five minutes was employed. When the trap door on the reactor was released resulting in instantaneous depressurization, the large coal particles were shattered into minute discrete particles of varying sizes (See Figure 3). The fractured coal particles were subjected to a Sp. Gr. 1.3 float-sink operation and the float and sink samples were analyzed by thermogravimetric analysis (TGA). A mechanically crushed sample to -100 mesh was also subjected to the sink-float and analyzed by TGA for comparison. The key observation is the fact that in the case of the CO₂ fractured coal the ash content of the float was only 2% whereas the sink has 29% ash and in the case of the mechanically crushed sample the ash content is evenly distributed in the float and sink fractions (See Figure 4). This supports the concept that the supercritical CO₂ is fracturing only the carbonaceous

fraction while leaving the mineral matter intact i.e selective liberation of mineral matter.

A set of of thirty explosive shattering experiments were performed by varying temperatures and pressures (Experiment nos. CF01 to CF83, Table 1) and the fractured coal particles were separated into 13 different fractions by sieving The mesh sizes used and the corresponding particle size range in which the fractured coal particles were separated into is shown in Table 2. The feed coal charged into the reactor had a average particle diameter of 7-8.2 mm (7000-8200 microns). Table 1 gives the pressure and temperature conditions for the runs. Runs at 1300 psia and 40°C yielded discrete fractured coal particles, the majority of which were in the 38-605 micron range (Figure 5). More material was present in the smaller particle size range i.e. 38-250 microns at 40°C than at 20°C. Ash analysis of the sieved fractions showed that the larger particle size fractions (700-4000 microns) contained as much as 50% of the ash (Figure 6).

As shown in Figure in the executive summary, a reduction in mean mass size of the fractured coal particles from 860 microns to 420 microns on going from 900 psia (subcritical) to 1300 psia (supercritical) at 40°C with 7-8.2 mm particle size feed vividly demonstrated the need to be operating under supercritical conditions. In the subcritical region, fracture was favoured at lower temperatures, while the higher (40°C) temperature was most effective in the supercritical region. The removal of water from the pores of the coal, before explosive shattering, adversely affected the fracture results. It appears that drying of the coal resulted in the collapse of some pore structures Mineral matter particle size distribution was determined in a coulter "multisizer" by burning off the carbonaceous matter in a oxygen plasma furnace at room temperature (Courtesy Dick Bourke, Allison Gas Turbines). Comparision of the mineral matter particle size before and after explosive shattering showed no significant differences (Figure 7 & 8). This showed that only the carbonaceous component of coal was exposively shattered into discrete particles as postulated by us.

Further experimentation showed that the degree of comminution is nearly the same in the temperature range of 29-59°C (with pressure at a constant 1500 psia). However, the runs at subcritical temperatures were not as good for

all the IBCSP samples. The increase in pressure has a more dramatic effect on the degree of comminution. On going form 900 psia to 1800 psia more fine coal is produced. Thus, 47.8% of IBCSP coal sample 2 passed through a 80 mesh seive at 1200 psia at 40°C, wheras at 1800 psia 62.1% of coal passed through the 80 mesh seive at the same temperature. The degree of comminution of IBCSP2, IBCSP6 is better than the other other two coal samples. Shattering of moisture free coal gave better comminution.

CONCLUSIONS

Coal particles can be effectively and selectively shattered into minuscule size particles using supercritical CO₂ while leaving the mineral matter intact. It was established that supercritical conditions are necessary for better comminution results. Temperature has very little effect on particle size distribution in the supercritical region, however increase in pressure has a very dramatic effect. An optimum pressure of 1800 psia and a temperature of 40°C was arrived at for the shattering experiment. Higher pressures would produce even finer coal.

RECOMMENDATIONS

The greatest potential and definitely the most exciting possibility for the project is the coupling of our CO₂ explosive fracture comminution process to the CO₂-coal slurry pipeline concepts. In fact, Southwestern Public Service Co. just finished a successful test run for the operation of a two mile, four feet diameter test loop carrying a coal-CO₂ slurry at its Harrington Station coal-fired power plant near Amarillo, Texas. Thus, the incorporation of a explosive fracture comminution step prior to the transportation would allow more clean coal to be transported.

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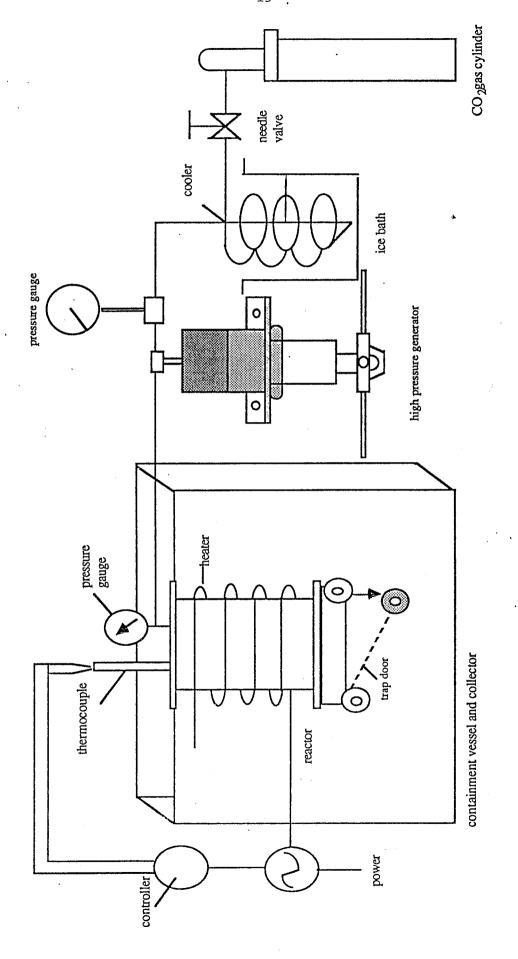
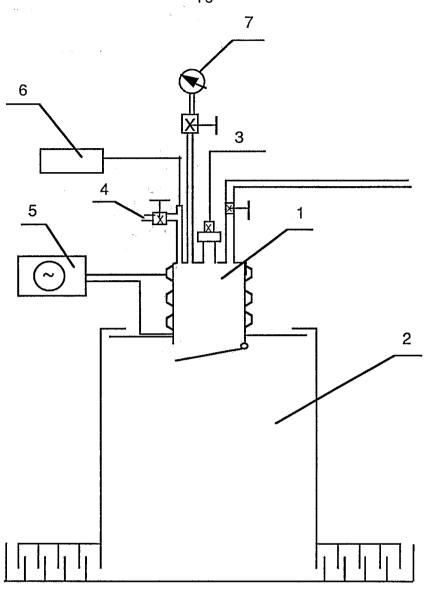


Figure 1. Experimental Setup for the Explosive Fracture Comminution of Coal Using Supercritical Calrbon Dioxide



- 1--Reactor
- 2--Collector
- 3--Coal inlet
- 4--Gas discharge
- 5--Heater
- 6--Thermometer
- 7--Pressure gauge

Figure 2. New Reactor Design with Containmnet & Collection Chamber

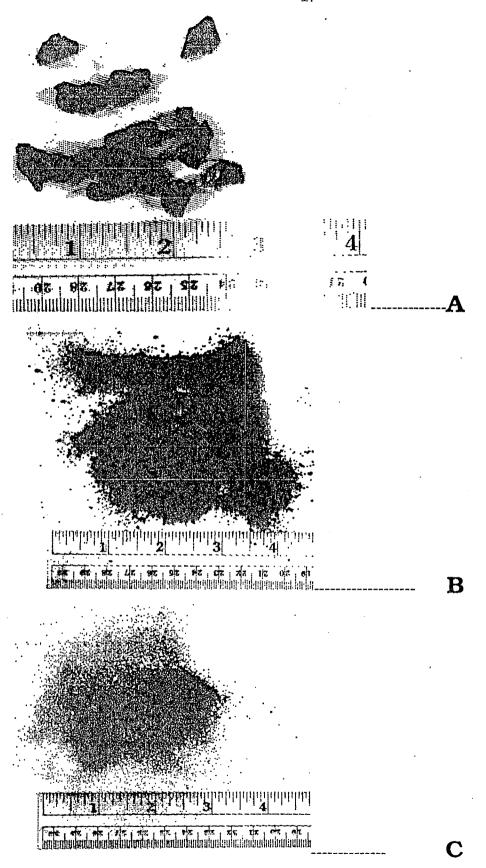


Figure 2. A) Starting coal particles. B&C) Fractured coal particles--different particle sizes

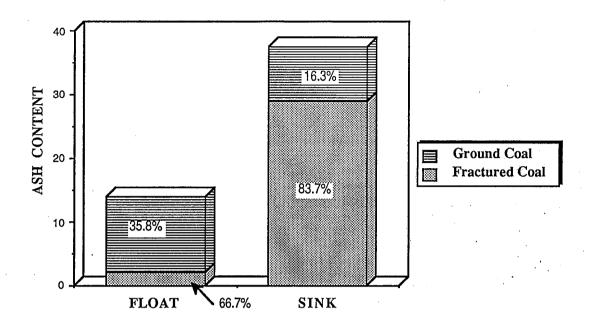


Figure 4. Ash Content of Float & Sink Fractions from Fractured and Mechanically Ground Coal

TABLE 1
COAL SHATTERING EXPERIMENTS

				Sieving
	Temp	Pressure		into 13
Code CF01 CF02 CF03 CF11 CF12 CF13 CF21 CF23 CF33 CF33 CF43 CF43 CF53 CF53 CF53 CF54 CF53 CF54 CF56 CF61 CF62	(C) 40 40 40 40 30 30 30 30 20 20 40 40 40 30 30 20 20 40 40 40 40 40 40 40 40	(psig) 1300 1300 1300 1300 1300 1300 1300 130	Shattering done done done done done done done done	Fractions done done done done done done done done
CF63	40	900	done	done
CF71	30	900	done	done
CF72	30	900	done	done
CF73	30	900	done	done
CF81	20	900	done	done
CF82	20	900	done	done
CF83	20	900	done	done

TABLE 2

Particle Size/Mesh Used for Fractionation

of Fractured Coal Particles

MESH SIZE	PARTICLE SIZE (MICRONS)	AVERAGE
5-7	4000-2800	3400
7-9	2800-2000	2400
9-12	2000-1400	1700
12-16	1400-1000	1200
16-25	1000-710	855
25-35	710-500	605
35-45	500-325	428
45-60	355-250	303
60-80	250-180	215
80-120	180-125	153
120-170	125-90	108
170-230	90-63	77
230	63-0	38

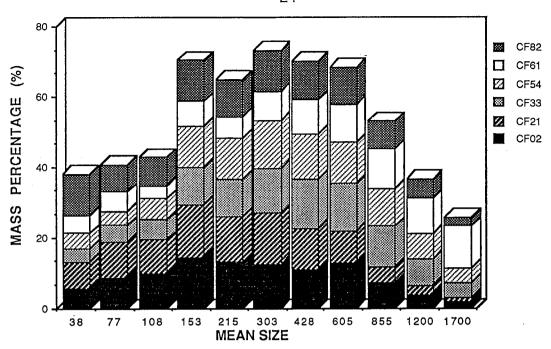


Figure 5. Comparison of the mass percent of fractured particles in different size fractions for a series of runs

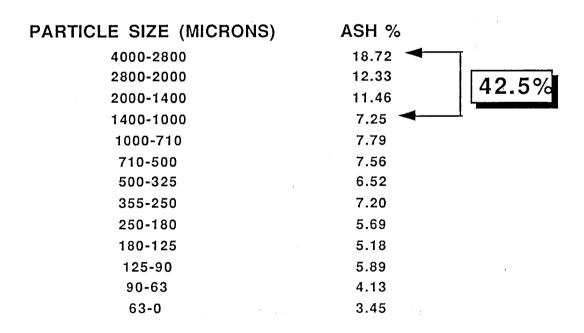


Figure 6. Ash analysis of sieved fractions

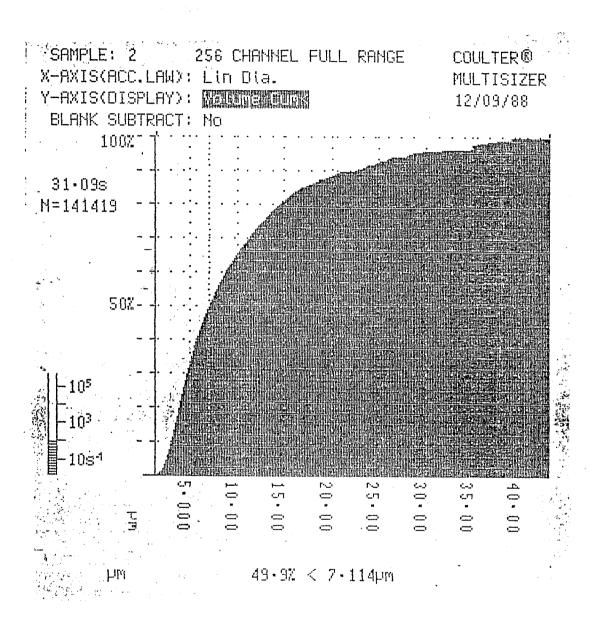


Figure 7. Particle Size Distribution of Starting Coal

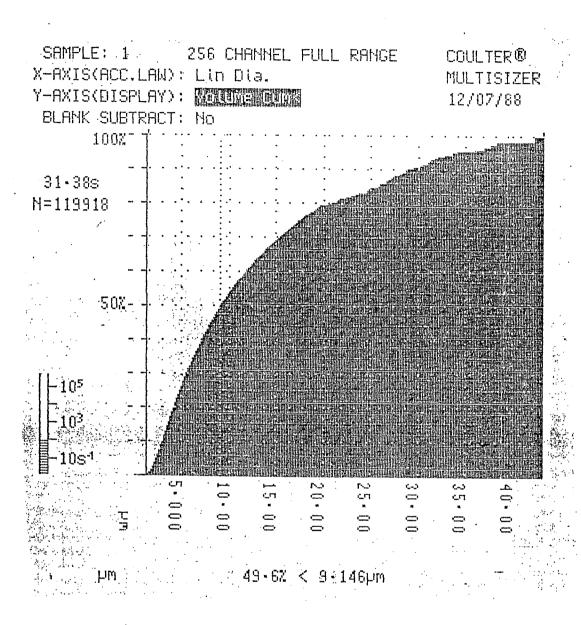


Figure 8. Particle Size Distribution of CO_2 Fractured Coal