Production of Superclean Coal by Wet-Grinding and Selective Flocculation

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ABSTRACT

The development of a method for removing both chlorine and sulfur from Illinois Basin coals is the objective of this project. The flow sheet established for sulfur and chlorine removal from coal includes four stages: (1) crush raw coal to -10 mesh, (2) use concentrating table to remove pyrite and other minerals, (3) wet-grind coal to an ultrafine particle size to liberate coal from pyrite and other minerals and to dissolve chlorine in process water, and (4) use selective flocculation to separate coal from mineral particles and chlorine-containing water. Three sets of experiments were conducted: (1) concentrating table tests, (2) selective flocculation tests, and (3) chlorine removal tests.

A concentrating table experiment on sample IBC-106 (9.0% ash and 3.77% sulfur) of the Illinois Basin Coal Sample Program (IBCSP) showed that pyritic sulfur was effectively reduced by this technique. Five fractions of the product were collected: clean (58.2% recovery), fine (35.9%), overflow (4.1%), middlings (1.0%), and heavy (0.8%). The pyritic sulfur content decreased from 1.85% in feed coal to 1.49%, 0.85%, and 0.82% in clean, fine, and overflow fractions, respectively.

Sample IBC-105 of the IBCSP (18.6% ash and 4.55% sulfur) was used in selective flocculation tests. The coal was ground in a stirred ball mill to generate a coal slurry with a particle-size distribution of 90% -19 μm. The slurry was dispersed at a high shear rate to make a complete suspension. Sodium metaphosphate was added as a dispersant. Various polymeric flocculants were tested to determine the selective flocculation performance of each. The acidity of the slurry (pH) and the flocculant dosage are critical in selective flocculation. When flocculant Calgon WCL-762 was used, the ash content decreased from 18.6% to 8.1% (56% reduction) in a single-stage flocculation process, and to 5.7% (69% reduction) in a two-stage process. The pyritic sulfur was reduced from 2.52% to 1.87% (26% reduction) in a two-stage process.

Chlorine reduction from coal by wet-grinding was investigated using sample IBC-109 (8.2 % ash, 1.13% sulfur, and 0.42% chlorine). Wet-grinding the coal to 90% -20 μm, and subsequently dewatering the slurry by selective flocculation reduced the chlorine content from 0.41% to within 0.11% and 0.15% (64%-74% reduction). In addition, ash content was reduced from 8.2% to within 4.0% and 5.4% (34%-51% reduction) and pyritic sulfur content from 0.50% to within 0.21% and 0.26% (42%-58% reduction) at 88% to 98% Btu recovery. Thus, this chlorine removal process can also remove significant amounts of ash and pyritic sulfur.

EXECUTIVE SUMMARY

High sulfur content in coal hinders the use of coal resources because sulfur dioxide emissions from utility and industrial boilers may cause acid rain. Chlorine in Illinois coal is another serious problem facing the Illinois coal industry because a high chlorine content is linked to boiler corrosion in coal combustion.

The chlorine content in Illinois coals increases with the depth of coal seams. Thus, coal mined underground has a higher chlorine content than surface-mined coal. At present, about 25% of the state's coal production contains 0.3% to 0.5% chlorine. The trend in future coal production in Illinois will be toward deeper underground mines, which will produce coal from seams in deeper parts of the basin known to have a high chlorine content. The chlorine problem will become more serious in the future. The purpose of this project is to develop a coal-cleaning process for removing both chlorine and sulfur from coal.

Chlorine Removal Process

The concept of this project evolved from an earlier ISGS project on chlorine removal from Illinois coal by wet-grinding coal to 80% -200 mesh (74 μm) and by subsequent leaching of the filter cake with hot water. To enhance chlorine removal and reduce pyritic sulfur and ash contents, we modified the previous process in the following aspects:

- tabling was used as an initial step to remove pyrite and other ash-forming minerals;

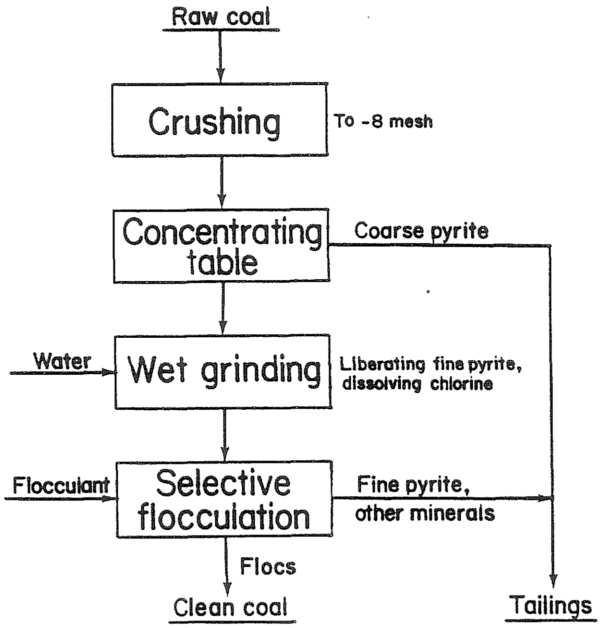

Figure 1 Flow sheet of the coal-cleaning process for removing sulfur and chlorine from Illinois coal.

- coal was wet-ground to finer particle sizes (about 90% -20 µm) to enhance the liberation of coal particles from minerals and increase the dissolution of chlorine in process water; and
- selective flocculation was used to remove the water from the slurry, which contains mineral particles in suspension and chloride in solution.

The process includes the following four stages (fig. 1).

- Crushing: Raw coal is crushed to -10 mesh.
- Wet concentrating table separation: Coarse pyrite particles in coal are removed by wet concentrating table technique.
- Wet-grinding: Raw coal is ground in a stirred ball mill (attritor) to 90% -20 µm. Coal particles are liberated from pyrite and other minerals, and chlorine is dissolved in process water.
- Selective flocculation: Fine pyrite particles and other ash-forming minerals (quartz, clay minerals, and calcite) are removed from coal.

Samples

Three samples from the Illinois Basin Coal Sample Program (IBCSP) were used in this study. Sample IBC-106 (9.0% ash, 3.77% sulfur, and 0.02% chlorine) was used for concentrating table tests because the sample was used extensively in the development of the ISGS aggregate flotation process, and tabling results may provide additional insights into the sulfur removability. Sample IBC-105 (18.6% ash, 4.55% sulfur, and 0.10% chlorine), collected and packaged under an inert atmosphere, is the least weathered among the samples of the IBCSP; it was used in selective flocculation tests. High-chlorine coal IBC-109 (8.2% ash, 1.13% sulfur, and 0.42% chlorine) was used in the chlorine removal study.

Concentrating Table Tests

Concentrating table separation was used to remove coarse pyrite from coal. Coal sample IBC-106 was ground to -10 mesh and then separated into four fractions on the table: clean, fine, middlings, and heavy. The overflow water from the fine fraction contained suspended coal, which was collected by filtering the water. The weight percentage of these five parts are 58.2% (clean), 35.9% (fine), 4.1% (suspended), 1.0% (middlings), and 0.8% (heavy). The clean product, which includes clean, fine, and suspended fractions, totals 98.2%. The reject, including middlings and heavy fractions, totals 1.8%. The ash content was reduced from 9.0 wt% in feed coal to 8.1 wt% in combined clean product (10% reduction) and pyritic sulfur content from 1.85 wt% to 1.23 wt% (33% reduction). Thus, the concentrating table technique is a useful initial step for removing coarse pyrite and other minerals.

Wet-Grinding

The coal sample was dry ground to -60 mesh and then wet-ground in a stirred ball mill. The slurry generated by wet-grinding IBC-105 in the mill for 12 minutes has a particle-size distribution of 90% -18.8 μm (fig. 2).

Selective Flocculation

The selective flocculation procedure consists of two major steps: dispersion and flocculation. The slurry was transferred into a 1000-mL beaker and diluted to a pulp density of 2%. Two dispersants, sodium metaphosphate (SMP) and polyacrylate xanthate (PAAX), were added to the slurry, and the mixture was stirred at a high shear rate to make a complete suspension. A polymeric flocculant was then added to aggregate the coal particles, which formed settling flocs while the minerals were kept in suspension in the supernate. Of prime importance to the performance of the selective flocculation process are properties of dispersants and flocculants, dosages of dispersant and flocculant, and acidity of liquid.

Polymeric flocculants from Calgon Corp. (WCL-762), Dow Chemical Co. (Separans), Allied Colloids Inc. (Percols), and American Cyanamid Co. (Superflocs) were tested to determine their selective flocculation performance. Sodium metaphosphate proved to be an effective dispersant for ash reduction. When the Calgon flocculant WCL-762 was used, a single-stage selective flocculation procedure reduced the ash content in coal IBC-105 from 18.6% to 8.1% (56% reduction), and a two-stage procedure reduced the ash content from 18.6% to 5.7% (69% reduction). The pyritic sulfur was reduced in the two-stage procedure from 2.52% to 1.87% (26% reduction).

Chlorine Removal

Chlorine removal experiments were conducted using two flocculants (WCL-762 and Percol 155) on sample IBC-109 (0.42% chlorine). The coal was dry-ground to -60 mesh and then wet-ground for 10 minutes in a stirred ball mill. The particle-size distribution of the slurry was 50% -8.5 μm and 90% -20.2 μm. A large portion of chlorine in the coal was dissolved in the process water during wet-grinding. The coal was then separated from the process water by selective flocculation.

Six selective flocculation tests were conducted using flocculant WCL-762. The chlorine content was reduced from 0.42% in raw coal to within 0.14% and 0.15% (64%-67% reduction). The ash content was reduced from 8.2% in raw coal to 4.0%-5.1% (38%-51% reduction), and the pyritic sulfur was reduced from 0.50% to between 0.21% and 0.23% (54%-58% reduction) at 88% to 94% Btu recovery.

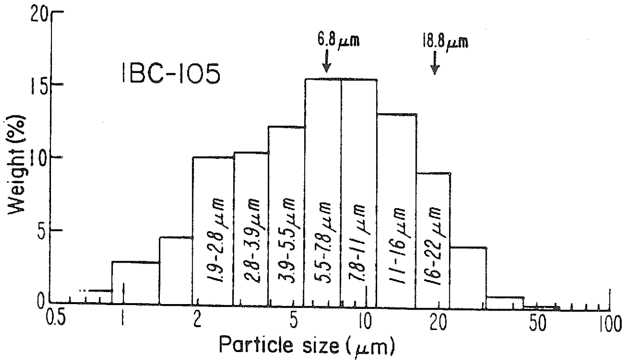

Figure 2 A typical particle-size distribution of slurry generated by wet-grinding coal in a stirred ball mill.

In four dewatering tests using flocculant Percol 155, the chlorine content was reduced from 0.42% in raw coal to between 0.11% and 0.15% (64%-74% reduction). The ash content was reduced from 8.2% to between 4.8% and 5.5% (33%-41% reduction), and the pyritic sulfur content was reduced from 0.50% to within 0.22% and 0.26% (42%-56% reduction) at 94% to 98% Btu recovery.

Conclusions

The flow sheet for chlorine and sulfur removal from coal includes four stages:

crushing, tabling, wet-grinding, and selective flocculation. The concentrating table step effectively reduced pyritic sulfur and ash contents. Wet-grinding coal in a stirred ball mill generated coal slurry suitable for selective flocculation. Ash content was reduced effectively from coal IBC-105 by selective flocculation but not the sulfur content. More work in preparing a pyrite-specific dispersant and a better understanding of changing surface properties of pyrite during the cleaning process are needed for achieving a higher pyrite rejection. Chlorine in coal IBC-109 was reduced markedly by wet-grinding the coal to ultrafine particle size (90% -20 μm) and dewatering the slurry by selective flocculation. Thus, an effective process for removing chlorine from Illinois coal has been developed. Another step for removing chlorine from the water, such as using ultrafiltration membrane technique, still needs to be developed to complete the chlorine-removal process.

OBJECTIVES

The purpose of this project was to develop a physical coal-cleaning process to enhance chlorine removal and reduce sulfur and ash in Illinois coal. Specific goals of the project were to:

- establish a flow sheet of a fine-coal cleaning process for removing chlorine and sulfur from Illinois coal;
- determine the extent that ash, sulfur, and chlorine can be reduced by wet-grinding and selective flocculation;
- conduct concentrating table tests to reduce the ash and pyrite contents in coal;
- enhance chlorine removal from coal by wet-grinding coal to ultrafine particle sizes;
- determine the effect of the properties of flocculants on the performance of selective flocculation process; and
- determine the effects of the pH of slurry, and dosages of dispersant and flocculant on ash and pyrite removal by selective flocculation.

INTRODUCTION AND BACKGROUND

In addition to having a high sulfur content, about half of the known, economically minable coal reserves in Illinois have a chlorine content above 0.3% (Chou 1990). Burning coal with a chlorine content at this level causes boiler corrosion in some cases, which has a negative impact on the marketability of high-chlorine coal. Therefore, we proposed to develop a physical cleaning process that removes both pyritic sulfur and chlorine from coal.

The extent that chlorine is removed by a physical cleaning method depends on the forms of chlorine in coal. The chlorine in Illinois Basin coal occurs in two chemical forms: chloride ion occurring as NaCl dissolved in pore water, and ionic chloride adsorbed on the inner surface of micropores in the organic matter (Chou 1990). In high-chlorine coal, most of the chlorine is in the adsorbed form.

The process to remove chlorine and sulfur involves four stages: (1) crushing raw coal to -10 mesh, (2) wet concentrating table separation, (3) wet-grinding to liberate coal particles from minerals and dissolve chlorine in the process water, and (4) selective flocculation.

We showed previously that wet-grinding coal to 80% -200 mesh (74 μm) and subsequent leaching filter cake with hot water is an effective process to remove chlorine from Illinois coal; more chlorine is removed when coal is ground to finer particle sizes (Chou 1989). In this study, coal is wet-ground to even finer particle size (about 90% -20 μm) to determine chlorine removability.

Grinding coal to such fine particle size also is needed for deep cleaning to remove pyrite and other minerals from coal. Because this particle size is much lower than the optimum sizes for froth flotation (50-140 mesh, 106-300 μm) (Zimmerman 1979), we proposed to use selective flocculation to remove water from slurry (dewatering). Selective flocculation also is effective for removing ash and pyritic sulfur from coal (Hucko 1977, Attia 1985, Attia 1986, Attia and Yu 1987, Attia et al. 1988, Elzeky et al. 1990, Ding and Erten 1990).

The mechanism of selective flocculation involves the process of coal particles forming aggregates (flocs) while mineral particles are kept in suspension. When a polymeric flocculant is mixed into the coal-water slurry, a polymer thread tends to adsorb onto a particle of coal. The thread may form a loop and the end of the thread may dangle in the liquid and become attached to another coal particle. Thus, a bridge develops between the two particles and large coal flocs may be formed. Removal of pyrite and other minerals from coal by selective flocculation depends on the difference in surface properties between coal and pyrite and other minerals.

EXPERIMENTAL PROCEDURES

Samples

Three coals from the Illinois Basin Coal Sample Program (IBCSP) were used in the study. Sample IBC-106 (9.0% ash, 3.77% sulfur) of the IBCSP was used for the concentrating table tests. Sample IBC-105 (18.6% ash, 4.55% sulfur) was used in selective flocculation tests because this coal was collected and packaged under an inert atmosphere, and thus was least weathered among the samples of the IBCSP. Sample IBC-109 (8.2% ash, 1.13% sulfur, and 0.42% chlorine) was used in the study of chlorine removal because it is the only high-chlorine coal in the IBCSP.

Equipment and Apparatus

We used the following equipment.

- Hazen-Quinn 10 in. by 6 in. dual crushing unit to reduce the particle size of coal to -10 mesh, and a stirred ball mill to wet-grind coal to prepare ultrafine coal slurry,
- Hazen-Quinn wet sample splitter to divide coal slurry into 12 fractions,
- Microtrac II Model 7997-10 particle-size analyzer from Leeds and Northrup Instruments to determine particle-size distribution in the range of 0.7 μm to 700 μm for coal,
- Deister shaking table,
- Ultra-Turrax T25 dispersing unit to provide high shear velocity in preparing coal-water suspension,
- Orion Expandable Ion Analyzer (EA940) with a pH electrode to determine pH
- Omega PHH-1X pH tester,
- Shimadzu UV-160 spectrophotometer, and
- AMRAY 1830 scanning electron microscope to examine the microstructure of coal flocs.

General Chemicals

The following chemicals were used.

- Carbon disulfide, CS_2, Fisher Scientific No. C184-500,
- Ethyl alcohol,
- Sodium hydroxide,
- Polyacrylic acid, $(CH_2CHCOOH)_n$, molecular weight 5000, 50% solid in water, Polysciences, Inc. catalogue No. 6519-250, lot No. 75198,
- Hydrogen chloride,
- Acetone, and
- Sodium metaphosphate, $(NaPO_3)_xNa_2O$, $x \cong 13$, Fisher Scientific No. S333-500.

Analytical Methods

Coal samples were analyzed at the ISGS Coal Analysis Laboratory using ASTM methods.

Concentrating Table Tests

Coal sample IBC-106, crushed to -10 mesh, was processed on the concentrating table to separate it into four fractions: clean, fine, middlings, and heavy. An additional fraction was also collected: suspended coal particles in the water that overflowed from the fine fraction.

Wet-Grinding Experiments

A coal sample (750 g) was transferred to a stirred ball mill. Then 1,450 mL of tap water and 50 mL of 1M sodium hydroxide were added to the mill. The mixture was ground for 10 to 12 minutes, generating a coal slurry with a particle-size distribution of about 90% -20 µm. The coal slurry was transferred into a container with 2 liters of tap water and then split into 12 fractions using a Hazen-Quinn wet-sample splitter. The particle-size distribution of coal slurry was determined on a Microtrac particle-size analyzer.

Selective Flocculation

The selective flocculation process is described in two major steps: dispersion and flocculation.

Dispersion The slurry was transferred into a 1,000-mL beaker and diluted to a pulp density of 2%. Two dispersants, sodium metaphosphate (SMP) and polyacrylate xanthate (PAAX), were added to the slurry and the mixture was stirred at a high shear rate to make a complete suspension.

Dispersants Sodium metaphosphate is used as a general dispersant for ash removal. A polyxanthate dispersant PAAX (polyacrylic acid/xanthate) (Attia 1985, 1986; Attia et al. 1988) was used for pyrite dispersion. It is prepared using the following procedures. Place 3 grams of polyacrylic acid (MW 5,000) into a 50-mL Erlenmeyer flask. Add 10-mL of 6M NaOH. Cool the flask in an ice slurry. Add 3 mL of CS_2 into the solution in a fume hood. Shake the mixture in the flask on a constant temperature bath at 15°C for 48 hours. Transfer the content to a centrifuge tube and centrifuge it for 3 minutes. Remove by pipetting the excess CS_2 at the bottom of the tube. Pour the solution into a 400-mL beaker. Wash the solution with 100 mL of ethyl alcohol. Discard the alcohol layer. Dissolve the orange polyxanthate precipitate in 20 mL of 1M NaOH. Store the product in a refrigerator.

Flocculation Polymeric flocculants were used to form coal aggregates (flocs) from coal particles dispersed in a liquid, while leaving mineral particles suspended in the supernate. Of prime importance to the performance of the selective flocculation process are the properties of flocculants, dosage of flocculant, and acidity of liquid. During the selective flocculation test, the acidity of the slurry was adjusted to a desired pH value by adding 0.1M NaOH or 0.1M HCl. Flocculant was added to the slurry as it was stirred. After 10 seconds, the slurry was stirred at a lower shear rate for 3 minutes. The slurry was let then stand for 5 minutes. The supernate was siphoned away. The flocs were filtered to remove remaining water and dried.

Table 1 Synthetic polymers for selective flocculation tests.

Company	Trade name	Type
Allied Colloids Inc.	Percol	polyacrylamide, polyacrylate, or copolymer of acrylate and acrylamide
American Cyanamid Co.	Superfloc	polyacrylamide, polyamines, or polyammonium quaternary salt
Calgon Corp.	(WCL-762)	(not available)
Dow Chemical Co.	Separan (AP30, AP273, MG200)	polyacrylamide

Flocculants The flocculants used are synthetic polymers from Allied Colloids Inc., American Cyanamid Co., Calgon Corp., and Dow Chemical Co. The trade names and polymer types are listed in table 1.

The charge and compounds of flocculants from Allied Colloids Inc. and American Cyanamid Co. are listed below:

Allied Colloids Inc.

Percol 352(cationic)	Low cationic copolymer of a quaternary acrylate salt and acrylamide, high molecular weight
Percol 455 (cationic)	Slightly cationic copolymer of a quaternary acrylate salt and acrylamide, high molecular weight
Percol 592 (cationic)	Medium cationic copolymer of a quaternary acrylate salt and acrylamide, very high molecular weight
Percol 155 (anionic)	Medium anionic copolymer of sodium acrylate and acrylamide, high molecular weight
Percol 156 (anionic)	Highly anionic copolymer of sodium acrylate and acrylamide, high molecular weight
Percol 358 (anionic)	Highly anionic acrylamide copolymer, high molecular weight
Percol 1011 (anionic)	Medium anionic copolymer of sodium acrylate and acrylamide, very high molecular weight
Percol E10 (anionic)	Low anionic, copolymer of sodium acrylate and acrylamide, very high molecular weight
Percol E24 (anionic)	Low anionic copolymer of sodium acrylate and acrylamide, moderately high molecular weight
Percol 611 (anionic)	Very highly anionic salt of a polymeric carboxylic acid (polyacrylate), high molecular weight
Percol 351 (nonionic)	Polyacrylamide, very high molecular weight

American Cyanamid Co.

Superfloc 310 (cationic)	Highly cationic polyamine in water
Superfloc 335 (cationic)	Highly cationic polyquaternary amine in water, high molecular weight
Superfloc 340 (cationic)	Highly cationic polyquaternary amine in water, high molecular weight
Superfloc 350 (cationic)	Highly cationic polyquaternary ammonium resin in water
Superfloc 360 (cationic)	Highly cationic polyquaternary ammonium resin in water, high molecular weight
Superfloc 362 (cationic)	Highly cationic polyquaternary ammonium resin in water
Superfloc 206+ (anionic)	Highly anionic polyacrylamide
Superfloc 208+ (anionic)	Highly anionic polyacrylamide
Superfloc 210+ (anionic)	Polyacrylamide, high molecular weight
Superfloc 212+ (anionic)	Polyacrylamide, high molecular weight

Scanning Electron Microscopic Observation of Coal Flocs

Four coal floc samples, FC-1, FC-2, FC-3, and FC-4, were prepared for SEM examination using the selective flocculation and flocculant Percol E10 in four dosages: 21.4 mg/L, 14.3 mg/L, 7.1 mg/L, and 3.6 mg/L (Test No. 0514-1, -2, -3, and -4), respectively. Some particles (1 to 10 μm) of pyrite, quartz, clay minerals, and calcite were observed. The flocs were more closely packed for samples FC-1 and FC-2 than for samples FC-3 and FC-4 because the former formed with a higher dosage of the flocculant than the latter.

RESULTS AND DISCUSSION

Concentrating Table Tests

Sample IBC-106 of the Illinois Basin Coal Sample Program was crushed to -10 mesh and then cleaned on a concentrating table. Five fractions were collected: clean (58.2%), fine (35.9%), overflow (4.1%), middlings (1.0%), and heavy (0.8%). Table 2 lists the analytical results of these fractions. The clean product, which includes clean, fine, and suspended fractions, totaled 98.2% of the sample. The reject, including middlings and heavy fractions, totaled 1.8%. The pyritic sulfur decreased significantly from feed coal (1.85% sulfur) to clean (1.49%), fine (0.85%), and suspended fractions (0.82%). The combined clean product of these three fractions has a weighted mean of pyritic sulfur content of 1.23% (33% removal), and shows a 10% ash reduction. The Btu recovery of the combined clean product is 98.7% (table 3).

Selective Flocculation Tests on Coal Sample IBC-105

All flocculants were used in selective flocculation tests at pH 10. Many flocculants need to be operated at pH levels lower than 10, except Separan MG200, Dow Chemical Co.; Percol 352 (low cationic), Percol 455 (slightly cationic), Percol E10 (low anionic), and Percol 351 (nonionic), all from Allied Colloids Inc.; and Superfloc 335, 340, 360, and 362 (all highly cationic polyquaternary amine in water), American Cyanamid Co.

WCL-762, Calgon Corporation Eleven selective flocculation tests on flocculant WCL-762 were conducted at pH levels between 6.0 and 7.7 (table 4). No flocculation occurred at higher pH

Table 2 Analyses of feed coal (IBC-106) and cleaning products, as well as recoveries and removabilities.

	Feed	Clean	Fine	Suspended*	Middlings	Heavy
		Analysis no.				
		C30713	C30714	C30715	C30716	C30717
Moisture	10.4	9.7	3.0	5.1	1.0	0.9
Volatile matter	39.6	40.0	41.0	38.0	32.2	27.9
Fixed carbon	51.4	50.6	53.0	51.4	35.1	17.3
Ash	**9.0**	**9.3**	**5.9**	**10.6**	**32.6**	**54.8**
Carbon	71.73	72.01	75.46	71.87	48.40	23.36
Hydrogen	4.85	5.21	5.48	5.17	3.68	1.71
Nitrogen	1.71	1.27	1.37	1.15	1.15	0.31
Oxygen	8.93	8.46	8.86	8.59	1.54	--
Sulfate Sulfur	0.01	0.12	0.02	0.00	0.17	0.23
Pyritic Sulfur	**1.85**	**1.49**	**0.85**	**0.82**	**10.78**	**23.45**
Organic Sulfur	1.91	2.09	2.04	1.84	1.67	0.43
Total Sulfur	3.76	3.70	2.91	2.66	12.62	24.11
Chlorine	0.02	0.08	0.07	0.06	0.06	0.07
Calorific value (Btu/lb)	13,231	13,080	13,679	12,980	9,214	5,397
Recoveries and removabilities**						
Product wt%		58.2	35.9	4.1	1.0	0.8
Calorific value		58.	37.	4.	0.7	0.3
		(0.)	(+1.)	(0.)	(-0.3)	(-0.5)
Ash		60.	24.	5.	4.	5.
		(+2.)	(-12.)	(+1.)	(+3.)	(+4.)
Pyritic Sulfur		**47.**	**16.**	**2.**	**6.**	**10.**
		(-11.)	**(-20.)**	**(-2.)**	**(+5.)**	**(+9.)**
Total Sulfur		57.	28.	3.	3.	5.
		(-1.)	(-8.)	(-1.)	(+2.)	(+4.)

Note: The middlings and heavy fractions are the reject. Concentrations are in wt% on dry basis except the moisture content.

* Suspended coal in the water overflowed from the fine fraction.

** Recoveries are in wt% of feed coal. The number in parenthesis is removability, which is the difference between the recovery and wt% of a cleaning product. + indicates enrichment. - indicates reduction.

Table 3 Analyses of the feed coal (IBC-106), the combined clean product, and the reject produced in a concentrating table experiment.

	Feed	Combined clean product*	Reject*
Moisture	10.4	7.1	1.0
Volatile matter	39.6	40.3	30.3
Fixed carbon	51.4	51.5	27.2
Ash	**9.0**	**8.1**	**42.5**
Carbon	71.73	73.3	37.3
Hydrogen	4.85	5.31	2.80
Nitrogen	1.71	1.30	0.78
Oxygen	8.93	8.61	--
Sulfate sulfur	0.01	0.08	0.20
Pyritic sulfur	**1.85**	**1.23**	**16.4**
Organic sulfur	1.91	2.06	1.1
Total sulfur	3.76	3.37	17.7
Calorific value (Btu/lb)	13,231	13,294	7,518
Recoveries and removabilities**			
Product wt%		98.2	1.8
Calorific value		98.7 (+0.5)	1.0 (-0.8)
Ash		**88 (-10)**	**9 (+7)**
Pyritic sulfur		**65 (-33)**	**16 (+14)**
Total sulfur		88 (-10)	8 (+6)

Note: Concentrations are in wt% on dry basis except the moisture content.
* Feed coal is sample IBC-106 of the IBCSP. Listed under the combined clean product are weight mean values of combined clean, fine, and suspended coal fractions. The reject represents weighted mean values of the middlings and heavy fractions.
** Recoveries are in wt% of feed coal. The number in parenthesis is removability, which is the difference between recovery and wt% of a cleaning product. + sign indicates enrichment. - sign indicates reduction.

Table 4 Selective flocculation using flocculant WCL-762 on coal IBC-105. Dispersants SMP (300 mg/L) and PAAX (200 mg/L) were added.

Run	pH	Dosage (mg/L)	Analysis no.	Yield (%)	Ash content (%)	Ash removal (%)
0530-2	6.0	13.4	C31127	70.4	11.1	-40.3
0531-2	6.2	18.8	C31128	65.3	8.8	-52.7
0605-1	6.3	5.4	C31129	68.4	10.8	-41.9
0605-2	6.1	10.7	C31130	60.9	9.0	-51.6
0605-3	6.1	16.1	C31131	65.1	8.9	-52.2
0605-4	6.1	21.4	C31132	65.8	8.7	-53.2
0712-1	6.5	5.4	C31230	66.9	8.7	-53.2
0712-2	6.7	5.4	C31231	56.6	8.2	-55.9
0712-3	7.1	5.4	C31232	52.2	9.5	-48.9
0716-1	7.5	5.4	C31233	40.3	9.7	-47.8
0716-2	7.7	5.4	C31234	70.6	9.3	-50.0

Table 5 Selective flocculation tests using varying amounts of dispersant SMP. Illinois coal IBC-105 and flocculant WCL-762 (10.7 mg/L) were used. The acidity of the slurry was adjusted to pH 7.0.

Run	SMP dosage (mg/L)	Analysis no.	Yield (%)	Ash (%)	Ash removal (%)	Pyritic sulfur (%)	Pyritic sulfur removal (%)
0815-1	0	C31301	96.8	14.7	-21.0	2.32	-8.0
0815-2	170	C31302	79.5	8.7	-53.2	2.14	-15.1
0815-3	300	C31303	78.5	8.5	-54.3	2.19	-13.1
0815-4	600	C31304	79.4	8.1	-56.5	2.13	-15.5
0815-5	900	C31305	81.7	8.5	-54.3	2.17	-13.9

Table 6 Two-stage selective flocculation test on Illinois coal IBC-105. Flocculant WCL-762 (10.7 mg/L) and dispersant (300 mg/L SMP) were added in each stage. The acidity of slurry was adjusted to pH 7.0.

Run	Analysis no.	Yield (%)	Ash (%)	Ash removal (%)	Pyritic sulfur (%)	Pyritic sulfur removal (%)
815-6	C31306	68.3	5.7	-69.4	1.87	-25.8

Table 7 Selective flocculation tests using flocculant Percol 592 on coal IBC-105. Dispersants SMP (300 mg/L) and PAAX (200 mg/L) were added.

Run	pH	Dosage (mg/L)	Analysis no.	Yield (%)	Ash content (%)	Ash removal (%)
0611-3	6.0	14.3	C31167	72.4	12.2	-34.4
0611-4	6.6	14.3	C31168	68.4	12.4	-33.3
0612-1	6.6	7.1	C31169	69.1	11.5	-38.2
0613-1	6.5	5.7	C31170	70.4	11.3	-39.2
0719-1	6.4	2.9	C31268	74.9	11.7	-37.1
0719-2	6.0	2.9	C31269	76.8	11.8	-36.6
0723-1	6.5	2.9	C31270	76.2	11.5	-38.2

values. The yields of clean coal were between 40.3% and 70.6%; ash reduction was between 40.3% and 56.0%. Variations of yield and ash removal are not correlated with flocculant dosage or pH value. No reduction of total sulfur content was observed in this set of experiments, and pyritic sulfur content was not determined.

The effect of varying amounts of dispersant sodium metaphosphate (SMP) on selective flocculation was tested using flocculant WCL-762. The results are shown in table 5. The tests (0815-2 to 0815-5) with dispersant dosages between 170 mg/L and 900 mg/L showed a great increase of ash and pyritic sulfur removal as compared with the test 0815-1 without SMP.

Two-stage selective flocculation Ash and pyritic sulfur reduction were increased when the selective flocculation procedure was repeated (table 6). The clean coal has 5.7% ash (69.4% reduction) and 1.87% pyritic sulfur (25.8% reduction).

Percol 592 (cationic copolymer), Allied Colloids Inc. Seven selective flocculation tests were conducted at pH 6.0 to pH 6.6 (table 7). No flocculation occurred at pH 7 or higher. In each test, 300 mg/L of SMP was added. The yield of clean coal was between 68.4% and 76.8%, and ash removal between 33.3% and 39.2%. The tests using lower flocculant dosage (2.9 to 7.1 mg/L) showed slightly higher ash removal (36.6% to 39.2%) than the tests using higher flocculant dosage (14 mg/L), which showed ash removal between 33.3 and 34.4%. No reduction in total sulfur content was observed and pyritic sulfur content was not determined.

Table 8 Selective flocculation tests using flocculant Percol 155 on Illinois coal IBC-105 at different pH levels of the slurry. Dispersants SMP (300 mg/L) and PAAX (200 mg/L) were added.

Run	pH	Dosage (mg/L)	Analysis no.	Yield (%)	Ash content (%)	Ash removal (%)
0527-1	6.1	146	C31111	94.1	16.9	-9.1
0527-2	7.0	36	C31112	93.4	15.6	-16.1
0527-3	8.0	36	C31113	80.3	12.0	-35.5
0527-6	8.0	22	C31114	81.6	12.0	-35.5
0527-7	8.0	7.3	C31115	85.5	10.9	-41.1
0527-4	9.0	36	C31116	79.6	11.6	-37.6

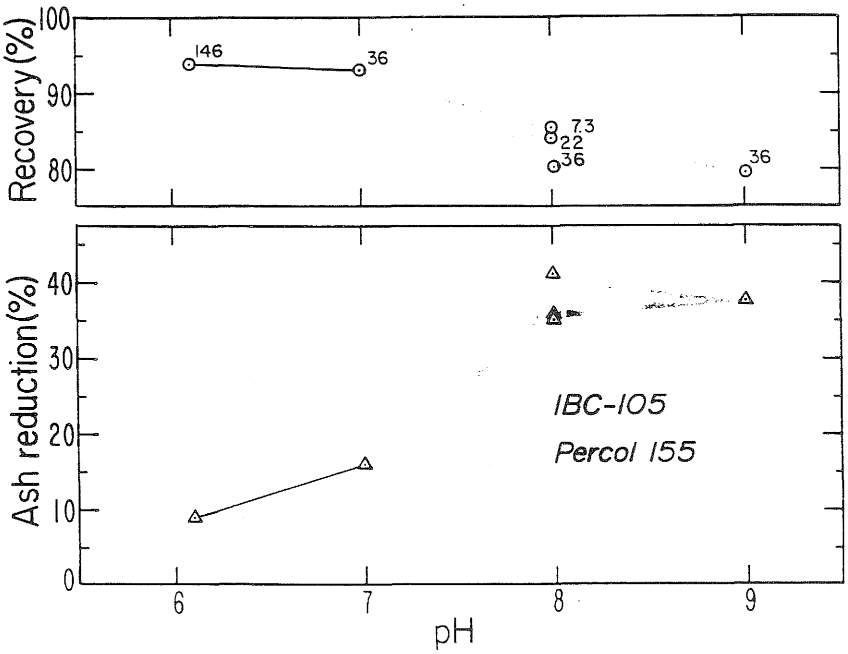

Figure 3 The yield of clean coal and ash reduction as a function of pH of the slurry in selective flocculation. Tests were conducted on Illinois Basin coal IBC-105 with flocculant Percol 155. The flocculant dosage of each test is indicated in the upper diagram.

Percol 155 (anionic copolymer), Allied Colloids Inc. In the test using Percol 155, flocculation occurred at pH levels from 6.1 to 9.0, but not at pH 10. Table 8 lists the test results. Figure 3 shows how the yield of clean coal and ash reduction vary with the pH level of the slurry. Higher ash removal was observed at pH 8 and pH 9 than at pH 6.1 and pH 7. Three tests at pH 8, each with a different flocculant dosage, indicate that the ash reduction was higher at a dosage of 7.3 mg/L (41.4% reduction) than at dosages of 22 mg/L and 36 mg/L (both 35.5% reduction). No reduction in total sulfur content was observed, and pyritic sulfur content was not determined.

Percol 156 (anionic copolymer), Allied Colloids Inc. Four selective flocculation tests using Percol 156 were conducted between pH 5 and pH 6.4 (table 9). No flocculation occurred at pH 7.0 or higher. The yields were between 65% and 85%, and ash reduction between 6% and 47%. A sharp decrease of ash reduction was observed when the acidity of the slurry decreased from

Table 9 Selective flocculation tests using flocculant Percol 156 on Illinois coal IBC-105. Dispersants SMP (300 mg/L) and PAAX (200 mg/L) were added.

Run	pH	Dosage (mg/L)	Analysis no.	Yield (%)	Ash content (%)	Ash removal (%)
0529-1	6.4	36	C31125	65	9.9	-47
0530-1	6.0	21	C31126	71	10.7	-42
0717-1	6.0	57	C31235	75	11.3	-39
0717-2	5.0	36	C31236	85	17.5	-6

Table 10 Selective flocculation tests using flocculant Percol 358 on Illinois coal IBC-105. Dispersants SMP (300 mg/L) and PAAX (200 mg/L) were added.

Run	pH	Dosage (mg/L)	Analysis no.	Yield (%)	Ash content (%)	Ash removal (%)
0606-3	5.7	21	C31162	74	11.0	-40.9
0607-4	6.0	5.7	C31166	77	12.0	-35.5
0607-2	6.2	2.9	C31164	72	11.3	-39.2
0607-1	6.1	1.4	C31163	75	11.5	-38.2
0607-3	6.1	0.7	C31165	68	10.0	-46.2

Table 11 Selective flocculation tests using flocculant Percol 1011 on Illinois coal IBC-105. Dispersants SMP (300 mg/L) and PAAX (200 mg/L) were added.

Run	pH	Dosage (mg/L)	Analysis no.	Yield (%)	Ash content (%)	Ash removal (%)
0620-1	6.0	14.3	C31191	81	14.6	-21.5
0620-2	6.6	14.3	C31192	80	13.5	-27.4
0620-3	7.1	14.3	C31193	73	11.6	-37.6
0621-2	6.7	4.3	C31195	83	13.4	-28.0
0621-1	6.8	1.4	C31194	80	12.6	-32.3
0621-3	6.8	0.7	C31196	77	11.8	-36.6

pH 6.0 to pH 5.0. No reduction in total sulfur content was observed, and pyritic sulfur content was not determined.

Percol 358 (anionic copolymer), Allied Colloids Inc. Five selective flocculation tests were conducted at pH 5.7 to pH 6.2. No flocculation occurred at pH 7.0 or higher. The results are listed in decreasing flocculant dosages (table 10) from 21 mg/L to 0.7 mg/L, which vary by a factor of 30. The yield only decreased slightly and ash removal increased slightly with decreasing flocculant dosage. No reduction in total sulfur content was observed, and pyritic sulfur content was not determined.

Percol 1011 (anionic copolymer), Allied Colloids Inc. Six selective flocculation tests were performed using Percol 1011 at pH 6.0 to pH 7.1 (table 11). No flocculation occurred at pH 8 or higher. For the first three tests using 14.3 mg/L of flocculant, the ash removal increased from 22% to 38% when the acidity of slurry changed from pH 6.0 to pH 7.1. Smaller flocculant dosages (0.7 to 4.3 mg/L) were used in the next three tests. The ash reduction increased from 28% to 37% with decreasing flocculant dosage from 4.3 mg/L to 0.7 mg/L. No reduction in total sulfur content was observed, and pyritic sulfur content was not determined.

Percol E10 (anionic copolymer), Allied Colloids Inc. Selective flocculation tests with varying flocculant dosages A series of six selective flocculation tests was conducted using different dosages of flocculant Percol E10, while keeping the pH constant (table 12). Dispersant sodium

Table 12 Selective flocculation tests using flocculant Percol E10 on Illinois coal IBC-105 . Dispersants SMP (133 mg/L) and PAAX (200 mg/L) were added.

Run	pH	Dosage (mg/L)	Analysis no.	Yield (%)	Ash content (%)	Ash removal (%)
0514-5	10.0	1.4	C31092	75.2	12.8	-31.2
0514-4	10.0	3.6	C31093	92.9	13.0	-30.1
0514-3	10.0	7.1	C31094	90.1	15.2	-18.3
0514-8*	10.0	7.1	C31095	91.5	14.7	-21.0
0514-2	10.0	14.3	C31096	92.2	16.9	-9.1
0514-1	10.0	21.4	C31097	88.7	17.3	-7.0

*No PAAX was added in this run.

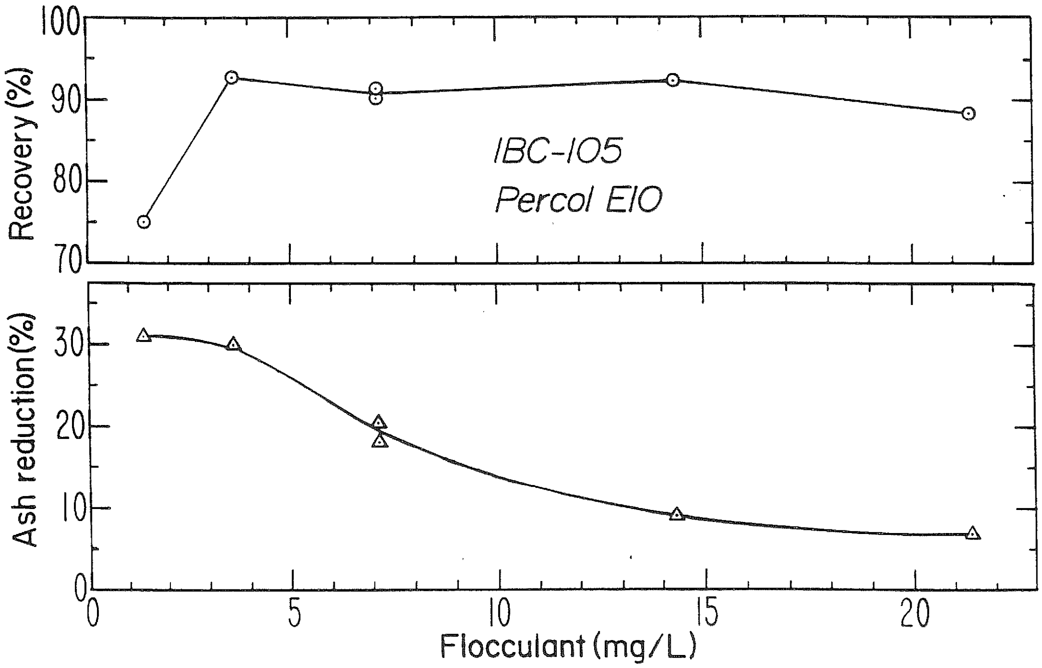

Figure 4 In a series of selective flocculation tests on Illinois Basin coal IBC-105, ash reduction increases as the dosage of flocculant Percol E10 decreases.

metaphosphate (133 mg/L) and PAAX were added. The percentage of clean coal recovery and ash reduction for these tests are shown in figure 4. The ash reduction increased from 7.0% to 31.2% with decreasing flocculant dosage from 21.4 mg/L to 1.4 mg/L, whereas the recovery varied between 75.2% and 92.9%. Use of a low flocculant dosage of 1.4 mg/L caused a high ash reduction of 31.2%, but a somewhat lower recovery of 75.2%. A flocculant dosage of 3.6 mg/L resulted in both high yield (92.9%) and ash reduction (30.1%). No reduction in total sulfur content was observed, and pyritic sulfur content was not determined.

Percol E24 (anionic copolymer), Allied Colloids Inc. Nine selective flocculation tests were made between pH 7.0 and pH 10.5 using flocculant Percol E24 (table 13). At the constant flocculant dosage, the ash reduction increased as the pH value increased, but ash reduction was low in all tests. No reduction in total sulfur content was observed, and pyritic sulfur was not determined.

Percol 611 (anionic polyacrylate), Allied Colloids Inc. Seven selective flocculation tests were conducted at pH 6.0 to pH 6.7 using flocculant Percol 611. No flocculation occurred at pH 8 and

Table 13 Selective flocculation tests using flocculant Percol E24 on Illinois coal IBC-105. Dispersants SMP (300 mg/L) and PAAX (200 mg/L) were added.

Run	pH	Dosage (mg/L)	Analysis no.	Yield (%)	Ash content (%)	Ash removal (%)
0625-2	7.0	14.3	C31197	85.9	17.5	-5.9
0626-1	7.0	14.3	C31198	83.4	17.0	-8.6
0626-2	7.5	14.3	C31199	84.1	16.1	-13.4
0627-1	8.0	14.3	C31200	83.5	15.5	-16.7
0627-2	8.5	14.3	C31266	81.5	13.9	-25.3
0627-3	9.1	14.3	C31201	80.7	15.1	-18.8
0627-4	9.5	14.3	C31267	83.7	15.4	-17.2
0627-5	10.0	14.3	C31228	--	15.6	-16.1
0702-1	10.5	14.3	C31229	74.9	14.4	-22.6

— not determined.

Table 14 Selective flocculation tests using flocculant Percol 611 on Illinois coal IBC-105. Dispersants SMP (300 mg/L) and PAAX (200 mg/L) were added.

Run	pH	Dosage (mg/L)	Analysis no.	Yield (%)	Ash content (%)	Ash removal (%)
0613-2	6.0	29	C31171	72	11.4	-38.7
0619-2	6.6	14	C31189	68	11.5	-38.2
0619-1	6.7	5.7	C31188	65	11.6	-37.6
0618-1	6.5	4.3	C31187	68	11.1	-40.3
0614-1	6.7	2.9	C31173	63	10.3	-44.6
0613-3	6.0	1.4	C31172	73	12.7	-31.7
0619-3	6.5	0.7	C31190	66	11.1	-40.3

Table 15 Selective flocculation tests using flocculant Percol 351 on Illinois coal IBC-105. Dispersants SMP (300 mg/L) and PAAX (200 mg/L) were added.

Run	pH	Dosage (mg/L)	Analysis no.	Yield (%)	Ash content (%)	Ash removal (%)
0509-4	11.3	3.6	C31081	54	14.7	-21.0
0509-3	11.4	7.1	C31082	72	15.8	-15.1
0509-2	11.1	14.3	C31083	87	15.5	-16.7
0404-1	(11)	21.4	C31059	83	17.2	-7.5
0509-5	11.3	21.4	C31084	95	18.4	-1.1
0509-1	11.1	28.6	C31085	99	18.3	-1.6
0509-6	10.1	35.7	C31086	100	19.1	+2.7

higher. The results are listed for flocculant dosages of 29 mg/L to 0.7 mg/L (table 14). Changing flocculant dosage by a factor of 41 did not influence the yield and ash removal. No reduction in total sulfur content was observed, and pyritic sulfur was not determined.

Percol 351 (nonionic polyacrylamide), Allied Colloids Inc. Seven flocculation tests using Percol 351 at various flocculant dosages were conducted at pH 11(table 15). As the flocculant dosage increased from 3.6 mg/L to 35.7 mg/L, the yield increased from 54% to 100%, but the ash removal decreased from -21.0% to +2.7%. Slight reduction of total sulfur content was observed in Run 0404-1 (-7.0%) and Run 0509-1 (-5.5%), but not in other runs. Pyritic sulfur content was not determined.

Superfloc 335 (cationic polyamine), American Cyanamid Co. Two flocculation tests using different dosages of Superfloc 335 were conducted at pH 11.5 (table 16). A lower dosage produced better ash and total-sulfur removal but lower yield.

Table 16 Selective flocculation tests using Superfloc 335 on Illinois coal IBC-105. The acidity of slurry was adjusted to pH 11.5.

Run	Dosage (mg/L)	Analysis no.	Yield (%)	Ash content (%)	Ash removal (%)	Sulfur content (%)	Sulfur removal (%)
0503-1	126	C31074	77.3	15.5	-16.7	4.40	-3.3
0504-1	70	C31075	70.8	12.0	-35.5	4.06	-10.8

Table 17 Selective flocculation tests using Superfloc 340 on coal IBC-105. Dispersants SMP (300 mg/L) and PAAX (200 mg/L) were added.

Run	pH	Dosage (mg/L)	Analysis no.	Yield (%)	Ash content (%)	Ash removal (%)
0724-2	11.0	5.7	C31271	52.8	13.5	-27.4

Table 18 Selective flocculation tests using Superfloc 362 on coal IBC-105. Dispersants SMP (300 mg/L) and PAAX (200 mg/L) were added.

Run	pH	Dosage (mg/L)	Analysis no.	Yield (%)	Ash content (%)	Ash removal (%)
0725-6	7.0	42.9	C31272	81.8	16.2	-12.9
0725-7	8.1	42.9	C31273	65.3	14.2	-23.7

Superfloc 340 (cationic polyamine), American Cyanamid Co. A selective flocculation test using Superfloc 340 was conducted (table 17); only moderate yield and low ash removal were obtained under the given conditions. The total sulfur content was essentially not changed (2.5% reduction).

Superfloc 362 (cationic polyammonium quaternary salt), American Cyanamid Co. Two selective flocculation tests using Superfloc 362 were conducted at two pH levels (table 18). Higher ash removal but lower yield were obtained at pH 8.1 than at pH 7.0. No reduction in total sulfur content was observed.

Summary on ash and pyritic removal on coal IBC-105 Significant ash reduction was observed in all experiments with SMP as a dispersant and various flocculants tested. For each flocculant, the ash removal and yield varied with changing pH value of slurry and flocculant dosage (tables 4 to 18). The maximum ash removal of each flocculant is listed in table 19. The highest ash removal was achieved using flocculant Calgon WCL-762: 56.6% ash reduction by a single-stage process and 69.4% reduction by a two-stage process.

Calgon WCL-762 and Superfloc 335 were the only two flocculants to cause significant reduction of pyritic sulfur content; no pyritic sulfur reduction was observed for other flocculants tested. In the test using WCL-762, a 15.5% pyritic sulfur reduction was achieved by a single-stage process (table 5, run 0815-4) and a 25.8% reduction by a two-stage process (table 6, run 0815-6). The test using Superfloc 335 produced a sulfur removal of 10.8% (table 16, run 0504-1).

Chlorine Removal from Coal

The chlorine-removal experiments were conducted on sample IBC-109 (0.42% chlorine). The coal was dry-ground to -60 mesh and then wet-ground for 10 minutes in a stirred ball mill. The particle-size distribution of the slurry was 50% -8.5 μm and 90% -20.2 μm (fig. 5). Chlorine in the coal was dissolved in process water during wet-grinding. The coal particles were separated from process water by selective flocculation. Two flocculants were used: WCL-762 and Percol 155.

Table 19 Summary of maximum ash removal achieved with each flocculant tested on Illinois coal IBC-105. Dispersants SMP (300 mg/L) and PAAX (200 mg/L) were added.

Flocculant	Run	pH	Dosage (mg/L)	Yield (%)	Ash content (%)	Ash removal (%)
WCL-762*	0815-4+	7.0	10.7	79.4	8.1	-56.5
WCL-762	0815-6§	7.0	10.7	68.3	5.7	-69.4
Cationic						
Percol 592	0613-1	6.5	5.7	70.4	11.3	-39.2
Anionic						
Percol 155	0527-7	8.0	7.3	85.5	10.9	-41.1
Percol 156	0529-1	6.4	36	65	9.9	-47
Percol 358	0607-3	6.1	0.7	68	10.0	-46.2
Percol 1011	0620-3	7.1	14.3	73	11.6	-37.6
Percol E10	0514-5	10.0	1.4	75.2	12.8	-31.2
Percol E24	0627-2	8.5	14.3	81.5	13.9	-25.3
Percol 611	0614-1	6.7	2.9	63	10.3	-44.6
Nonionic						
Percol 351	0509-4	11.3	3.6	54	14.7	-21.0
Cationic						
Superfloc 335	0504-1	11.5	70	70.8	12.0	-35.5
Superfloc 340	0724-2	11.0	5.7	52.8	13.5	-27.4
Superfloc 362	0725-7	8.1	42.9	65.3	14.2	-23.7

*Calgon Corp.
+SMP dosage 600 mg/L.
§Two-stage process.

Dewatering using flocculant WCL-762 Six selective flocculation tests were conducted using flocculant WCL-762. The results are listed in table 20. The chlorine content was reduced from 0.42% in raw coal to within 0.14% and 0.15% (64% to 67% reduction) in clean coal. The ash content was reduced from 8.2% in raw coal to between 4.0% and 5.1% (38%-51% reduction), and the pyritic sulfur was reduced from 0.50% to within 0.21% and 0.23% (54%-58% reduction) at 88% to 94% Btu recovery. The sulfur content in sample C31247 is equivalent to 0.7 lb SO_2/MBtu, a compliance coal with a low chlorine content.

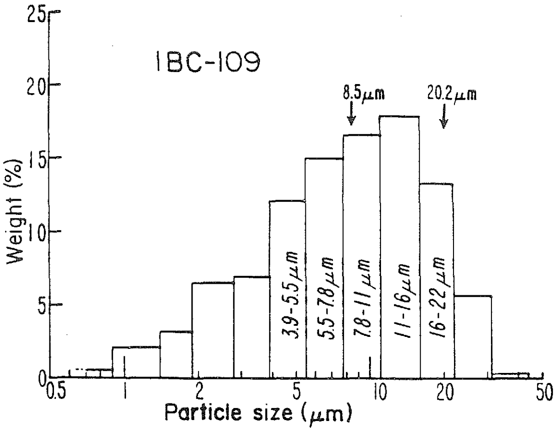

Figure 5 Particle-size distribution of IBC-109 (50% - 8.5 μm and 90% -20.2 μm) after dry-grinding to 60 mesh and wet-grinding for 10 minutes in a stirred ball mill.

The coal was ground on July 19, 1990. The first four runs were completed on the same day of coal grinding. The last two runs were conducted on August 10, 22 days after grinding. Similar results obtained on the two dates suggested that the storage of slurry for about 3 weeks did not significantly change selective flocculation behavior of the slurry.

Dewatering using flocculant Percol 155 Four selective flocculation tests were conducted using flocculant Percol 155. The results are listed in table 21. The chlorine content was reduced from 0.42% in raw coal to between 0.11% and 0.15% in clean coal (64%-74% reduction). The ash content decreased from 8.2% to within 4.8% and 5.5% (33%-41% reduction), and the pyritic sulfur content was reduced from 0.5% to between 0.22% and 0.26% (42%-56% reduction).

Table 20 Selective flocculation using flocculant WCL-762 to dewater the slurry in chlorine removal tests on Illinois coal IBC-109 (8.2% ash, 1.13% total sulfur, 0.50% pyritic sulfur, and 0.42% chlorine). Dispersant SMP (300 mg/L) was added.

Test	Analysis no.	pH	Dosage (mg/L)	Btu recovery (%)	Ash (%)	Total sulfur (%)	Pyritic sulfur (%)	Chlorine (%)
0719-4	C31247	7.2	29	88	4.0 (-51)	0.91 (-19)	0.22 (-56)	0.14 (-67)
0719-5	C31248	7.3	57	88	4.3 (-48)	0.92 (-19)	0.22 (-56)	0.15 (-64)
0719-6	C31249	6.3	29	94	5.1 (-38)	0.93 (-18)	0.23 (-54)	0.15 (-64)
0719-7	C31250	6.0	57	91	4.8 (-41)	0.94 (-17)	0.21 (-58)	0.15 (-64)
0810-1	C31288	7.0	29	—	4.6 (-44)	0.90 (-20)	—	—
0810-2	C31289	6.2	29	—	4.8 (-41)	0.90 (-20)	—	—

Note: The percent removal is shown in the parenthesis. Detailed analysis of samples C31247 to C31250 are listed in table 22.
— not determined.

Table 21 Selective flocculation using flocculant Percol 155 to dewater the slurry in chlorine removal tests on Illinois coal IBC-109 (8.2% ash, 1.13% total sulfur, 0.50% pyritic sulfur, and 0.42% chlorine). Dispersant SMP (300 mg/L) was added.

Test	Analysis no.	pH	Dosage (mg/L)	Btu recovery (%)	Ash (%)	Total sulfur (%)	Pyritic sulfur (%)	Chlorine (%)
0719-1	C31245	7.1	5.7	98	5.4 (-34)	0.95 (-16)	0.22 (-56)	0.15 (-64)
0719-2	C31246	8.0	5.7	94	5.3 (-35)	0.96 (-15)	0.26 (-42)	0.11 (-74)
0810-3	C31290	7.1	1.7	—	5.5 (-33)	0.93 (-18)	—	—
0810-4	C31291	8.0	1.7	—	4.8 (-41)	0.91 (-19)	—	—

Note: The percent removal is shown in the parenthesis. Detailed analyses of samples C31245 and C31246 are listed in table 22.
— not determined.

CONCLUSIONS AND RECOMMENDATIONS

(1) A flow sheet for chlorine and sulfur removal from coal was established. The operations include crushing, tabling, wet-grinding, and selective flocculation.

(2) Concentrating table experiments showed that pyritic sulfur and ash contents were effectively reduced. Thus, tabling is a useful initial step for removing coarse pyrite and other mineral particles.

(3) Wet-grinding in a stirred ball mill generates a coal slurry suitable for selective flocculation.

Table 22 Compositions and calorific values (in Btu/lb) of clean coal samples in chlorine removal tests on Illinois coal IBC-109.

	Analysis no.					
	C31245	C31246	C31247	C31348	C31249	C31250
Moisture	2.5	2.2	7.0	8.4	4.5	3.2
Volatile matter	37.6	37.6	38.8	38.9	38.0	38.1
Fixed carbon	56.9	57.0	57.1	56.9	56.9	57.0
Ash	**5.4**	**5.3**	**4.0**	**4.2**	**5.1**	**4.8**
Carbon	76.24	76.60	77.13	77.09	76.54	76.76
Hydrogen	4.16	4.45	4.42	4.46	4.28	4.20
Nitrogen	1.44	1.65	1.71	1.62	1.74	1.61
Oxygen	11.79	11.03	11.80	11.66	11.44	11.70
Pyritic sulfur	**0.22**	**0.26**	**0.22**	**0.22**	**0.23**	**0.21**
Sulfate sulfur	0.041	0.051	0.054	0.044	0.052	0.052
Organic sulfur	0.69	0.65	0.64	0.66	0.65	0.68
Total sulfur	**0.95**	**0.96**	**0.91**	**0.92**	**0.93**	**0.94**
Chlorine	**0.15**	**0.11**	**0.14**	**0.15**	**0.15**	**0.15**
Calorific value	13,551	13,480	13,800	13,824	13,656	13,694

Note: Concentrations are in wt% on dry basis except the moisture content.

(4) Polymeric flocculants from Calgon Corp. (WCL-762), Dow Chemical Co. (Separans), Allied Colloids Inc. (Percols), and American Cyanamid Co. (Superflocs) were tested to determine their selective flocculation performance. Each polymeric flocculant has characteristic flocculating capacity and properties.

(5) Experiments indicate that the acidity of the slurry and the flocculant dosage are the most critical factors controlling the performance of selective flocculation.

(6) Sodium metaphosphate is an effective dispersant for ash reduction.

(7) Dispersant PAAX, synthesized in our laboratory, was used in selective flocculation tests. Pyritic sulfur was not removed effectively in the tests on sample IBC-105. Future research should be directed to preparing effective pyrite-specific dispersants and understanding the change of surface properties of pyrite and coal during the process of selective flocculation.

(8) Flocculant Calgon WCL-762 used in a single-stage selective flocculation reduced the ash content in coal IBC-105 from 18.6% to 8.1% (56% reduction), and to 5.7% (69% reduction) by a two-stage process. The pyritic sulfur decreased from 2.52% to 1.87% (26% reduction) in a two-stage process.

(9) Ultrafine wet-grinding to 90% -20 µm and subsequent dewatering by selective flocculation markedly reduced the chlorine content of Illinois coal IBC-109 from 0.42% to between 0.11% and 0.15% (64%-74% reduction). This process also reduced the ash content from 8.2% to between 4.0% and 5.5%, and pyritic sulfur from 0.50% to between 0.21% and 0.26%. Thus, we have developed an effective process for chlorine removal from 0.42% to within 0.1% and 0.15% level.

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