

INCREASING REACTIVITY OF ILLINOIS COAL FOR USE IN IGCC PROCESSES

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INTRODUCTION

Integrated gasification combined cycle (IGCC) processes are expected to become an essential component for energy production in the next century. IGCC processes are able to utilize high sulfur coal and typically achieve power generation efficiencies in excess of 40%. The Illinois State Geological Survey (ISGS) has played an important role in the commercialization of IGCC technology in the Illinois Basin region [1]. A 400 ton test of Illinois coal initiated by the ISGS and performed with Destec Energy, Inc. at the Dow Chemical Plaquemine IGCC facility [2] in 1990, showed for the first time that Illinois coal, a caking coal, could be used effectively in this process. Continued use of Illinois coal at this facility, however, was not possible since the sulfur recovery unit at this plant was designed for low sulfur coal. The results from the 400 ton test, however, were used to help design the The Wabash River Coal Gasification Repowering Project in Terre Haute, IN, initially a joint venture between Destec Energy, Inc. and PSI Energy, Inc. and currently owned and operated by Dynegy Corporation. This ongoing commercial demonstration began operation in August 1995 and currently gasifies up to 2,500 tons/day of an Indiana coal to produce 262 MW of power [3]. The Wabash River facility is the first of its kind to utilize bituminous coal having moderate amounts of sulfur (1-2%). The sulfur in the coal is recovered as elemental sulfur, a saleable byproduct.

In the majority of IGCC processes (e.g., Texaco, Shell), coal is gasified in a one stage process at relatively high temperatures (1400°C) in an oxygen rich environment. In such a process, the coal delivered to the gasifier is converted to gaseous products within seconds, therefore, the intrinsic reactivity of the coal is not an important issue since mass transfer considerations dictate how fast the coal reacts. However, in a two-stage process such as the one being commercially demonstrated by Dynegy, coal is also gasified at lower temperatures (900-1000°C) in a mostly reducing atmosphere in the second stage gasifier. Leftover heat from the oxygen blown gasifier is recovered in the second stage by gasifying additional coal, up to 30% of the total feed. Since complete char burnout is rarely achieved in this second stage, the reactivity of the coal fed into the second stage becomes an important process consideration. If partially reacted char from the second stage is reintroduced into the first stage, this could lower the overall efficiency of the process since high ash, low BTU char now replaces some of the low ash, high BTU feed coal. The development of IGCC technology that utilizes a two stage process to gasify coal provides an opportunity to optimize the reactivity of the coal added to the second stage gasifier. A more reactive coal and resultant char would lead to higher levels of conversion in the second stage gasifier and more efficient operation. The use of more reactive coals in IGCC processes should lead to even more efficient production of electricity [4, 5].

Proven methods for improving the gasification reactivity of coal include adding a catalyst, e.g., calcium, to the coal. Preoxidation of coal may also increase its gasification reactivity. Tar formation, which happens to be detrimental to IGCC process performance, could be suppressed by preoxidation as well as by the addition of calcium to the coal. The objective of this study [6, 7] was to evaluate and improve the gasification behavior of Illinois coal for use in two-stage IGCC processes. This commercial application precludes the use of alkali metal catalysts such as sodium or potassium since these may volatilize and damage the gas turbines. In this paper, we discuss various methods to increase char reactivity through coal preoxidation and/or addition of a suitable catalyst such as calcium or iron.

EXPERIMENTAL

Coal/Char Preparation

Coals were obtained from the Illinois Basin Coal Sample Program (IBCSP) [8] and from selected coal mines throughout Illinois. An Indiana coal currently being used in an IGCC two-stage gasifier was also studied. Coal chars were prepared from these coals in a 2 in. ID horizontal tube furnace (N₂, 900°C, 30°C/min, 0.5 h). After pyrolysis, agglomerated chars were ground with a mortar and pestle to a -100 mesh particle size. Some coals were preoxidized in air at 225°C for 1 h prior to gasification tests and/or catalyst addition.

Catalyst Addition

Calcium (acetate) and iron (chloride) were added to as-received coal by ion-exchange (IE) at different pHs. IE was conducted by shaking 250 mL of each metal solution with 1 g of coal for 24 h, after which the samples were filtered and the solids submitted for metal analysis. Ca and Fe were added by IE at their natural pH (5.5 and 2.0, respectively) or at pH = 10 by dropwise addition of NaOH. Gypsum was added to 250 ml of H₂O containing 20 g of preoxidized coal and stirred at 60°C for 8 h. The pH of this mixture was adjusted to pH = 11.

Coal/Char Gasification Reactivity

Two experimental procedures (isothermal and nonisothermal) were used to measure the gasification reactivity of the char or coal. The specific char gasification reactivity (g C/g C/h) as a function of char conversion (X_c) in 1 atm CO_2 at 850-940°C was determined by isothermal thermogravimetric analysis (TGA). The coal gasification rate as a function of time in 0.80 atm $\text{CO}_2/0.20$ atm H_2O was determined by nonisothermal TGA (25-1000°C, 100°C/min). A Cahn TGA (TG-131) was used to monitor changes in char or coal reactivity with conversion or time. A typical isothermal experiment proceeded as follows. A char sample (10 mg) was placed in a platinum pan suspended from the Cahn weighing unit and enclosed by a Vycor hangdown tube. High purity nitrogen (99.999%) was introduced into the system (150 cm^3/min) and allowed to displace for 0.5 h the air in the reaction chamber. After flushing the system with N_2 , the temperature was raised to 120°C and held for 15 minutes to record the dry weight. The sample was then rapidly heated (100°C/min) to the reaction temperature. Within 1 minute after reaching the setpoint, the temperature stabilized and the initial weight was recorded. High purity carbon dioxide (99.999%) was then introduced into the reaction chamber at the same flow rate (150 cm^3/min). Sample weights were recorded continuously at 30 s intervals until the run was terminated at 90-95% conversion. An ash determination was made for each sample by switching from the reactant gas to oxygen at the reaction temperature.

RESULTS AND DISCUSSION

Char Reactivity

Figure 1 presents typical reactivity profiles (specific gasification rate, R_p , versus conversion, X_c) for IBC-101 char gasified in 1 atm CO_2 at 850-940°C. Activation energies calculated from the Arrhenius plots for this char (Figure 2) increase slightly from 55.0 to 63.8 kcal/mol, well within values reported in the literature [9] for coal char gasification in CO_2 . Thus, the reactivity profiles obtained for IBC-101 under these conditions are considered to be in the chemically controlled regime and represent true variations in char reactivity with conversion.

The gasification reactivities of chars prepared from nine of the twelve coals in the IBCSP were determined in 1 atm CO_2 at 850 by isothermal thermogravimetric analysis. Chars were prepared in a tube furnace under identical pyrolysis conditions (N_2 , 30°C/min, 900°C, 0.5 h) prior to the gasification tests. Figure 3 presents reactivity profiles for these coals and two other Illinois coals, Cedar Creek and Crown II coal, and the Indiana coal. Figure 3 shows that the reactivities of IBC-103, IBC-105, IBC-106 and IBC-108 were comparable over the entire conversion range, while IBC-101, IBC-107 and IBC-109 were most reactive and the high ash IBC-104 char and Indiana coal were the least reactive. Cedar Creek coal having more than twice the sulfur content (the more sulfur the better since sulfur can be recovered as a saleable byproduct), but less ash than the Indiana coal, was more than twice as reactive as the Indiana coal char. Char made from Crown II coal was nearly four times as reactive as the Indiana coal char. Another method of measuring reactivity involving rapid heating (100°C/min) of the coal sample in a reducing atmosphere (80% CO_2 , 20% H_2O , 1 atm) was also used. The results from nonisothermal tests confirmed the order of reactivity obtained from these isothermal tests.

Effect of Preoxidation

Perhaps the most cost effective way to increase the reactivity of bituminous coal would be to add oxygen to it prior to gasification either by natural weathering or a low temperature oxidation treatment. A recent study [10] has shown improved combustion performance for naturally weathered (oxidized) coals versus deep mined (unoxidized) coals. The pretreatment of coal with oxygen is thought to promote the cross linking reactions between aromatic units in the coal structure preventing their rearrangement during pyrolysis (melting) and increasing the surface area of the resultant char, which could lead to an increase in char reactivity [11]. Figure 4 presents reactivity profiles for several as-received and preoxidized coals. Preoxidation of IBC-102 and Cedar Creek coal results in little or no increase in reactivity. Preoxidation of the Indiana coal actually leads to a decrease in reactivity. Coal preoxidation also reduces the BTU content of the coal, which would decrease overall process efficiency. Thus, coal preoxidation alone does not appear to be a viable means to increase the gasification reactivity of these candidate coals for the Wabash River gasification plant.

Catalyst Addition

Calcium and iron are appropriate catalysts for the strict corrosivity requirements of IGCC processes. Abotsi et al. [12] recently evaluated the uptake of iron by coal particles dispersed in water. A surfactant, sodium dodecylsulfate, was used to increase the dispersion and stability of iron loaded onto an Illinois #6 coal. Iron loadings up to 3 weight percent were achieved. Adsorption of iron and calcium onto coal occurs through exchange of the cations (Fe^{+2} , Ca^{+2}) with the protons on the carboxylic or phenolic acidic groups on coal. The use of a surfactant promotes the dissociation of the surface functional groups, which creates a negative charge on the surface of the coal. This will favor adsorption of Fe^{+2} through electrostatic interaction between Fe^{+2} and negatively charged active sites. Practically speaking, efficient catalyst adsorption and dispersion in the coal would need to occur during transport of a coal-water slurry to the gasifier.

The effects of calcium and iron on the gasification behavior of Illinois coal were examined. Calcium and/or iron were added to IBC-101, Crown II, IBC-102, and Indiana coal by ion exchange at various pHs (2.2, 5.6 and 10) to determine to what extent these catalysts would impact char reactivity. Figure 5 presents reactivity profiles (conversion versus time) for Ca- and Fe-catalyzed IBC-101 chars gasified in 1 atm CO₂ at 850°C. When Ca is added by IE at pH = 10, there is a three-fold increase in reactivity. Iron added by IE at pH = 2.2 (natural pH) is seen to have little impact on reactivity. On the other hand, Fe added by IE at pH = 10 increases reactivity by more than a factor of two. These results indicate the importance of using an alkaline medium for preparing Ca- and Fe- catalyzed coals by IE. To the best of our knowledge, no one has shown that adjusting pH during catalyst loading can enhance subsequent reactivity of the catalyzed char. Figure 6 presents reactivity profiles for Crown II coal chars gasified in 1 atm CO₂ at 850°C. The as-received Crown II coal was our most reactive Illinois coal (Figure 3). When Ca or Fe is added to this coal by IE (at pH = 10), char reactivity again increases by more than a factor of two. The reactivities of these coals prepared at their natural pH (either 5.6 or 2.2) are significantly lower than those at pH = 10, consistent with results obtained for the catalyzed IBC-101 coals (Figure 5). Figure 6 presents reactivity profiles for the Indiana coal char. Addition of Ca and Fe at pH = 10 leads to only a slight increase in reactivity. Addition of Ca at its natural pH (5.6) actually decreases reactivity. These results indicate that Illinois coals are not only more reactive than the Indiana coal, but also respond better to the catalyst treatments perhaps due to a more favorable surface chemistry, i.e., greater amount of carbon-oxygen groups that facilitate ion-exchange between the catalyst and the carbon surface.

Effect of Coal Preoxidation on Catalytic Activity

Crown II and IBC-102 coal were preoxidized prior to catalyst addition to determine the effect, if any, of preoxidation on catalytic activity. Although preoxidation in itself was not an effective means to increase char reactivity (Figure 4), coal preoxidation prior to catalyst addition by IE would likely provide additional ion exchange sites for calcium or iron to react with. The coal samples were preoxidized by exposure to air at 225°C for 90 min before catalyst addition by ion exchange using calcium acetate. Figure 8 shows the effects of preoxidation and pH on reactivity profiles of Ca-catalyzed IBC-102 chars gasified in 1 atm CO₂ at 850°C. The reactivity of preoxidized IBC-102 coal is similar to that of the as-received coal. When Ca is loaded by IE at pH = 11 the maximum rates for both the Ca-catalyzed raw coal and preoxidized coal chars (between 6.5 and 8.3 g/g/h) are significantly higher than that of the raw coal (1 g/g/h). The increase in reactivity is more pronounced for the preoxidized sample (8.3 g/g/h). Both Ca-catalyzed chars maintain the high reactivity over most of the conversion range ($X_c = 0.15 - 0.80$). In addition, the Ca-catalyzed chars prepared at their natural pH (5.6) was not as reactive as the one prepared at pH = 11 indicating once again the importance of pH during catalyst preparation.

Figure 9 shows the effects of preoxidation and pH on the reactivity profiles obtained for Ca-catalyzed Crown II chars gasified in 1 atm CO₂ at 850°C. The preoxidized Crown II coal was less reactive than the as-received coal. When Ca is loaded by IE at pH = 11 on the as-received and preoxidized Crown II coal, the gasification rates of both chars increase dramatically. The Ca-catalyzed preoxidized Crown II char maintains its high reactivity to higher levels of conversion compared to the Ca-catalyzed as-received coal indicating better dispersion of the catalyst in the preoxidized sample. The increases in reactivity are not as dramatic when the catalyst is loaded at its natural pH = 5.6, which is consistent with the trends observed for IBC-102 coal (Figure 8). From the data presented in Figures 8 and 9, it appears that oxidation of the coal prior to catalyst addition had a positive effect on the reactivity of both IBC-102 and Crown II coals. Figure 10 shows non isothermal reactivity profiles for raw, preoxidized, Ca-catalyzed raw, and Ca-catalyzed preoxidized IBC-101 coal. Less volatile matter is given off during the pyrolysis stage (24-45 min) for the preoxidized coal and the reactivity of the raw and preoxidized chars are comparable (56-68 min). The Ca-catalyzed chars begin to gasify at a much earlier temperature (about 200°C) than the uncatalyzed chars. The catalyzed preoxidized char produces less volatile matter during the pyrolysis stage than the catalyzed raw coal, which would be expected based on the reactivities of uncatalyzed raw and preoxidized chars.

Use of Gypsum as a Catalyst Precursor

Although calcium acetate is an effective catalyst precursor, its cost may be too high to justify its use in IGCC processes. We also attempted to load calcium onto Crown II coal by ion exchange using gypsum (calcium sulfate) instead of calcium acetate. Figure 11 shows clearly the catalytic effect imparted by gypsum to preoxidized Crown II coal at pH = 11. This result was rather unexpected given the relatively low solubility of gypsum in water (2 g/L) versus calcium acetate (> 80 g/L). Gypsum-catalyzed char is about one half as reactive as calcium acetate-catalyzed char and several times more reactive than the preoxidized coal char. One would expect very little increase in reactivity based on the solubility of gypsum, however, it appears that a significant amount of calcium was incorporated into the coal. This probably occurs because gypsum continues to solubilize as calcium is ion exchanged onto the coal, thus maintaining a sufficient driving force for the ion exchange process. This so-called "shuttling" of calcium from gypsum to coal leads to substantial loadings of calcium given a sufficient amount of time. An increase in the amount of soak time from 8 h to say 48 h as well as optimizing pH of the solution could further increase calcium loadings achieved by gypsum. Figure 12 presents non isothermal reactivity profiles for gypsum-catalyzed chars prepared at pH = 5.6 and pH = 11. The catalyzed coal prepared at pH = 11 is significantly more reactive than the raw coal or catalyzed coal prepared at pH = 5.6. Volatile matter production of the pH = 11 char during pyrolysis is also noticeably less than that of the other two chars. The effects of pH (11

versus 5.6) and catalyst precursor (gypsum versus calcium acetate) on the reactivity of Crown II coal char are shown in Figure 13. Calcium acetate loaded coal is still more reactive than gypsum loaded coal. Perhaps an increase in soak time and/or a more suitable pH will narrow the gap between CaSO₄ and CaAc. Figure 14, shows the effect of calcium precursor on so-called tar formation or volatile matter evolution. Gypsum catalyzed coal produces significantly less volatile matter (part of which is comprised of tars) than the calcium acetate loaded coal

Possible benefits to be derived from using gypsum instead of other catalyst precursors include: 1) a cheap, readily source of catalyst is made available, 2) a new use is found for gypsum, a coal combustion by-product, 3) sulfur from the gypsum could be recovered as a valuable by product in post gasification cleanup processes, 4) calcium oxide would be converted to calcium carbonate, thus reducing emissions of carbon dioxide from the gasifier, and 5) calcium would suppress tar formation, which is detrimental to the performance of some gasifier systems. Dynegy adds limestone to the coal water slurry prior to gasification to improve the slagging behavior of the ash. The limestone, however, is essentially inert with respect to increasing reactivity. Along these same lines, SASOL, the operator of the largest single train gasification plant in the world, is experimenting with adding catalysts to their feed coal to increase coal throughput in their process [13]. SASOL is using a waste stream from a power plant that contains low concentrations of calcium and sodium as the catalyst precursor solution. Note that the calcium originates from gypsum present in the power plant waste water. An up to three-fold increase in reactivity was observed when their feed coal was allowed to soak in this solution [13]. SASOL operates eighteen fixed-bed Lurgi gasifiers to produce chemicals by Fischer-Tropsch synthesis.

Potential Use of Illinois Coal in IGCC Processes

Table 1 presents key gasification properties of both Illinois and Indiana coals. The % ash, % sulfur, heating value of some of the more promising coals in these two states shows that several Illinois coals have more favorable properties than the Indiana coal presently being gasified at the Wabash River gasification plant. Cedar Creek coal apparently has the most favorable combination of properties with respect to ash content (6.00%), sulfur content (3.8%) and heating value (12,271 BTU/lb). Table 1 also lists the gasification rates at 20% char conversion in 1 atm CO₂ at 850°C. The Crown 2 coal is seen to be most reactive Illinois coal, while Pattiki is the least reactive. The Indiana coal is the least reactive among all the coals listed. Another important property of the coal is its T₂₅₀ value, which is based on the composition of the ash and gives a good indication of its slagging behavior. These values have been calculated for each coal using a graphical correlation [14] and are also listed in Table 1. Any coal having a T₂₅₀ value less than 2350°F can be gasified without having to add limestone. Dynegy typically adds limestone to their coal/water slurry feed to modify the slagging behavior of the ash. If little or no limestone needs to be added, a significant savings in operating cost could be realized. It is interesting to note that the T₂₅₀ of the Industry mine coal is less than 2150, which is quite favorable for use in the Wabash River IGCC process. It is also important that the mineral matter content of the coal feed be as low as possible. Low ash coal means less waste disposal; less energy is required to heat the coal since ash also contains water that needs to be vaporized. The sulfur content of the Indiana coal is relatively low (1.71%) compared to what would work best in the sulfur recovery system. A coal with twice the sulfur content would be easily handled by the Dynegy system. Several tests with coals other than the one presently being using are planned in the near future. The coals to be tested, however, have not been decided. High sulfur (6%) petroleum coke is also being tested.

Table 1. Comparison of Illinois and Indiana coals.

	% ash	% S	T ₂₅₀ (°F)	R _{sp} ¹ (h ⁻¹)	BTU/lb ²	FSI
Illinois coals						
IBC-101	10.40	4.40	---	0.25	10,764	3.8
IBC-106	8.90	3.80	---	0.13	11,796	4.3
IBC-112	10.80	2.80	---	---	12,425	6.5
Crown 2	8.72	4.14	2,500	0.35	11,022	3.5
Industry Mine	7.07	3.58	< 2,150 ³	0.16	11,500	2.5
Cedar Creek	6.00	3.68	< 2,630 ³	0.16	12,271	3.0
Monterey 2	9.63	4.20	2,500	0.14	11,183	4.0
Old Ben 25	9.36	3.12	2,460	0.12	12,163	4.5
Pattiki	9.27	3.13	2,410	0.09	11,959	4.0
Eagle 2	9.57	3.32	2,420	---	11,867	4.5
Indiana coal	10.53	1.71	---	0.06	10,805	---

¹ char gasification rate at 20% conversion in 1 atm CO₂ at 850°C.

² corrected for equilibrium moisture.

³ the predicted temperature is below the temperature of critical viscosity, thus the graphical correlation used is not applicable, actual T₂₅₀ is less than the given temperature.

SUMMARY

The goal of this study was to provide Dynegy Corporation (formerly Destec Energy) with sufficient data to perform a large scale test with Illinois coal, either as received or modified by preoxidation and/or catalyst addition. An analyses of thirty four coals obtained from active mines throughout Illinois showed that several had lower ash content, higher sulfur content and heating value than the Indiana coal presently used by Dynegy. The Indiana coal char was the least reactive of all the chars tested except one made from high ash IBC-104 coal. An Illinois coal having considerably less ash (6.00%), more sulfur (3.68% S) and a higher heating value (12,271 BTU/lb) was about four times more reactive than the Indiana coal. Coal preoxidation had minimal effect on gasification reactivity of Illinois and Indiana coals. Coal reactivity was enhanced by adding catalysts (Ca or Fe) under conditions simulating those of a coal/water slurry. Catalytic effects were strongly dependent on the pH (> 10) of the ion exchange medium. Alkaline pH was found to be most effective. Three- to five-fold increases in reactivities as a function of pH (2.2 to 10) were observed for Ca- and Fe-loaded Illinois coals. Although coal preoxidation was not an effective means to increase char reactivity, preoxidation of the coal prior to catalyst addition enhanced the reactivity of the Ca-catalyzed coal by providing additional ion exchange sites. The use of gypsum (CaSO_4), a coal combustion by-product, instead of calcium acetate (CaAc), as a catalyst precursor was studied. The reactivity of gypsum-catalyzed char was significantly greater than that of uncatalyzed char, but less than one prepared using calcium acetate. The catalytic effect imparted by gypsum was surprising given that gypsum has a relatively low solubility compared to calcium acetate. Further work is needed to modify and test Illinois coals for use in IGCC processes. Binary catalyst systems, which have shown considerable promise in lower temperature gasification systems may provide superior performance relative to equivalent loadings of a single catalyst. Large scale tests need to be performed using ton quantities of optimized Illinois coal under process conditions simulating those used in the Dynegy entrained flow gasifier. The possibility of using Illinois coal in other IGCC processes such as the one to be demonstrated at the Grand Tower Power Station built in Southern Illinois [15] also needs to be examined.

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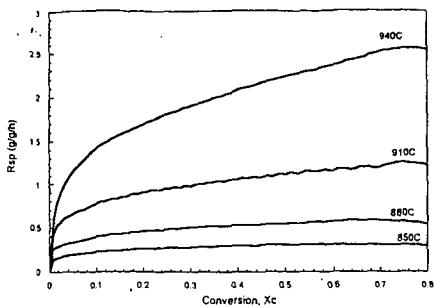


Figure 1. Reactivity profiles for IBC-101 char gasified in 1 atm CO₂.

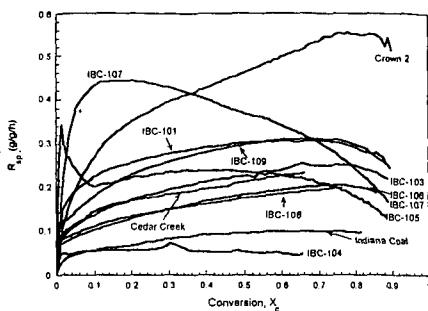


Figure 3. Reactivity profiles for Illinois and Indiana coal chars gasified in 1 atm CO₂ at 850 °C.

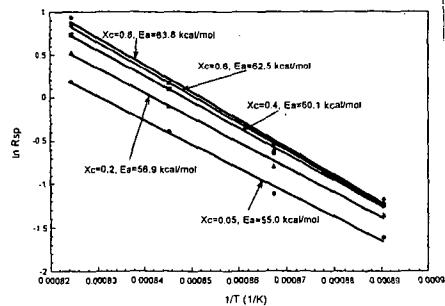


Figure 2. Arrhenius plots for IBC-101 char.

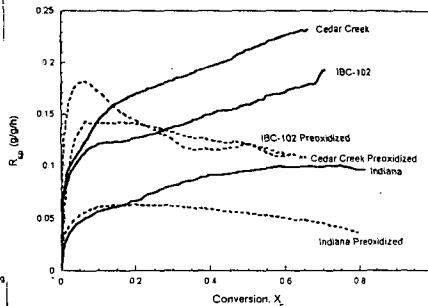


Figure 4. Effect of preoxidation (air, 225 °C, 1h) on gasification reactivity.

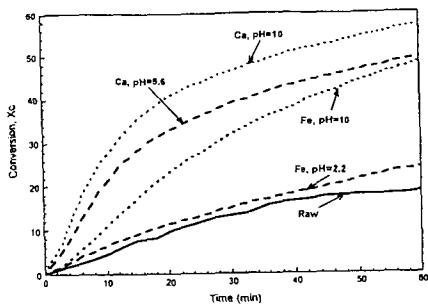


Figure 5. Effect of pH and catalyst on gasification reactivity of IBC-101 char.

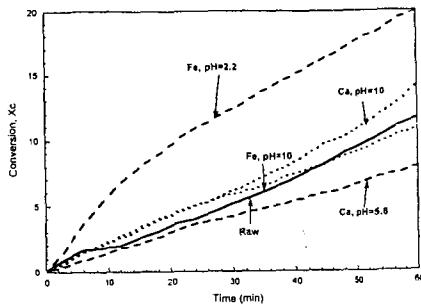


Figure 7. Effect of pH and catalyst on gasification reactivity of Indiana char.

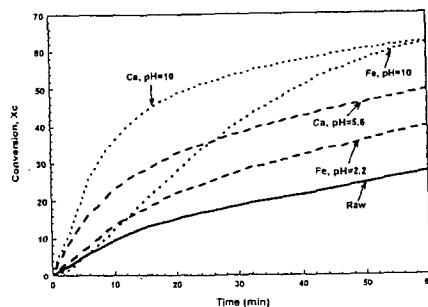


Figure 8. Effect of pH and catalyst on gasification reactivity of Crown II char.

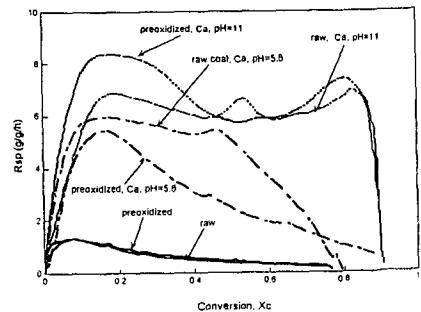


Figure 6. Effect of preoxidation and pH on reactivity of Ca-catalyzed IBC-102 char.

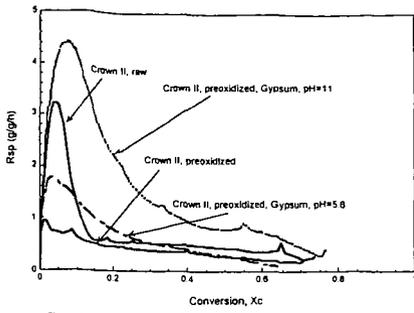


Figure 11. Use of gypsum (CaSO_4) as catalyst precursor.

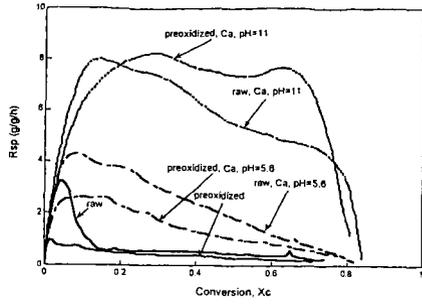


Figure 9. Effect of preoxidation and pH on reactivity of Ca-catalyzed Crown II char.

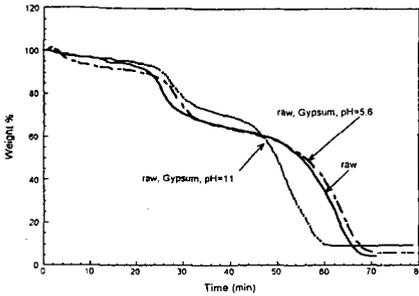


Figure 12. Effect of gypsum and pH on non isothermal reactivity of raw IBC-102 coal.

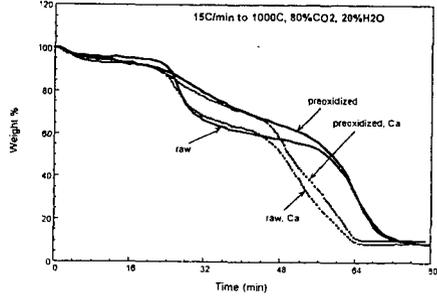


Figure 10. Effect of preoxidation and calcium addition on non isothermal reactivity of IBC-101 coal.

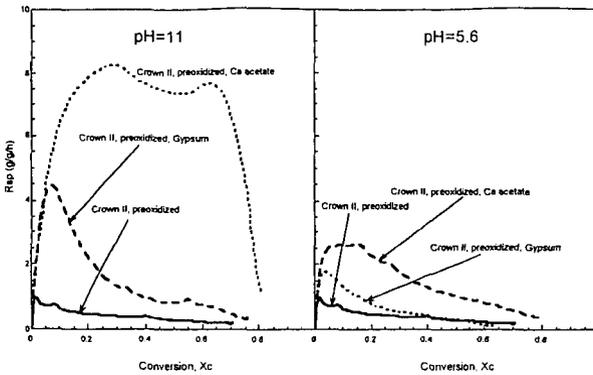


Figure 13. Comparison of gypsum and calcium acetate.

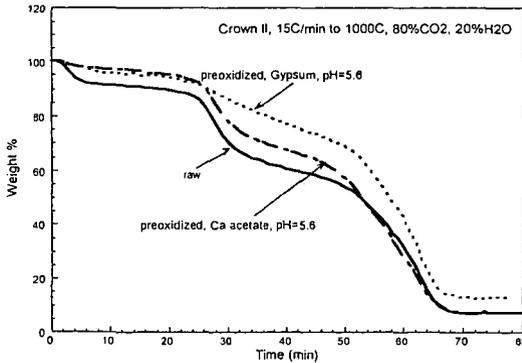


Figure 14. Effect of catalyst precursor on tar formation.