STUDY OF PORE STRUCTURE AND REACTIVITY OF
CANADIAN COAL-DERIVED CHARS

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ABSTRACT

The pore volume, surface area, compressibility and reactivity of 12 chars derived from Canadian coals ranking from lignite to anthracite, have been determined by mercury porosimetry, gas adsorption-desorption with N_2 and CO_2 adsorbates, helium and mercury densities and thermogravimetric technique. A comparison of the pore structure between the chars and their parent coals was made based on the results obtained from these techniques. The reactivity of chars was correlated with their physical and chemical properties. It has been found that a single relationship exists between the helium density and the carbon content of the coals and their chars. The chars are more porous but less compressible when compared with their parent coals.

(Keywords: coal; char; physical properties; reactivity)

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ÉTUDE DE LA STRUCTURE DES PORES ET DE LA RÉACTIVITÉ DES PRODUITS DE CARBONISATION DÉRIVÉS DES CHARBONS CANADIENS

par

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RÉSUMÉ

Le volume des pores, la valeur de la surface, la compressibilité et la réactivité de douze produits de carbonisation dérivés des charbons canadiens dont le rang variait de lignite à anthracite, ont été déterminés à l'aide de la porosimétrie à mercure, l'adsorption et la désorption avec du N_2 et CO_2 comme adsorbats. Des relations ont été dérivées, en outre, à partir des densités d'hélium et de mercure par la méthode thermogravimétrique. Les résultats de l'étude de la structure des pores des produits de carbonisation et des charbons d'origine obtenus par différentes techniques ont été comparés et étudiés. On a établi une corrélation entre la réactivité des produits de carbonisation et leurs propriétés physiques et chimiques. Il a été trouvé que la relation établie antérieurement entre la densité de l'hélium et la teneur en carbone des charbons pouvaient être utilisée avec les produits de carbonisation après avoir apporté une correction pour la teneur en minéraux.

Mots-clés: Charbon, produit de carbonisation, propriétés physiques, réactivité

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INTRODUCTION

The depletion of oil reserves is a future worldwide problem. Through research and development, alternative energy sources are being sought. One potential alternative receiving increased interest is the direct or indirect conversion of coal or biomass into liquid fuels. The commercial production of gasoline from coal in South Africa proves that this technology is practical, though it may not be economical based on current oil prices. Recently the IGCC (Integrated Gasification Combined Cycle) process has emerged as one of the most promising new technologies for coal-based electric power generation. The efficiency of any coal conversion depends largely on the coal reactivity which is greatly influenced by its chemical and physical properties.

In a previous publication[1], it has been shown that the reactivity of 11 Canadian coals varies with their pore structure and coal rank. Chars derived from 8 Canadian coals have also been studied at CANMET with respect to the gasification kinetics and the impact on char reactivity by the parent coal rank and by the CaO and MgO contents of the chars[2]. However, the pore system of these chars remains to be examined. The objective of this study was to determine the surface area and pore structure of 12 chars and the effect of these parameters on the char reactivity.

EXPERIMENTAL

MATERIALS

The four Canadian coals used to prepare additional chars consisted of Onakawana (lignite) from Ontario, Bienfait (lignite) from Saskatchewan, Tulameen (subbituminous) from Alberta and Mt. Klappan (anthracite) from British Columbia. The devolatilization procedure was similar to that used to prepare the first batch of 8 chars[2] except coal samples (particle size 3-6 mm) were heated at a rate of 5°C/min under a flow of N_2 at 2 L/min until a temperature of 900°C was reached. Tables 1 and 2 show the chemical analyses of the four coals and their derived chars. The analyses of the other 8 coals and their chars were reported previously[2]. For physical measurements the particle size of the char samples was controlled at 425-212μm (40-70 US standard mesh).

REACTIVITY MEASUREMENTS

The reactivity of char samples was determined thermogravimetrically using a Cahn electrobalance at 500°C with air as a gasification medium. A detailed procedure and the apparatus used have been described in an earlier publication[2].

APPARATUS

The mercury densities and pore volumes in the macropore (2.98-0.06 μm) and mesopore (0.06-0.0036 μm) ranges were determined using a commercial mercury porosimeter (Micromeritics Autopore 9200). The surface tension of mercury and contact angle were assumed to be 0.485 Nm^{-1} and 130°. The helium density measurement was obtained using a helium pycnometer Micromeritics 1302. The N_2 and CO_2 sorption studies were conducted using a Carlo Erba Sorptomatic 1822. For N_2 sorption (77K), the equilibration time was automatically controlled such that negligible change in pressure was observed over a 4 min period for each pressure point. For CO_2 adsorption (195K) the equilibration time was constantly set for 1 h. Equilibrium pressure (P) was gradually increased until it reached 30% or more of the saturation pressure (P_o).

The N_2 surface area and pore size distribution were determined using the BET equation and a procedure developed by Barett, Joyner and Halenda[3] in which the apparent pore radius was calculated by the Kelvin equation[4]. An effective cross-sectional area of 0.162 nm^2 was assumed for the N_2 molecule.

The CO_2 surface area was calculated using both the BET equation and the Dubinin-Radushkevich-Kaganer equation[5] (DRK equation, sometimes referred to as Dubinin-Polanyi equation) in which the concept of micropore filling in the Dubinin-Radushkevich (DR) equation[6] was replaced by surface coverage. Naturally, the DR equation could be used to calculate the micropore volume. The CO_2 effective cross-sectional area and the saturation vapor pressure of CO_2 at 195 K were assumed to be 0.195 nm^2 [7] and 186 kPa [8] respectively.

RESULTS AND DISCUSSION

Char is a major product derived from pyrolysis of coal and thus its property is greatly influenced by the constituents of its parent coal. The extent of change in property depends on the nature of the coal and the devola-

tilization procedure used[9]. Under the fixed condition described previously[2], the eight Canadian coal-derived chars exhibited some gasification behaviour which can be correlated with the chemical properties of their parent coals. Further investigation on the physical properties of 12 chars originated from 5 coal ranks has been conducted. The chemical and physical properties of these chars in relation to their reactivities are discussed in the following sections.

The char samples were first characterized by the mercury porosimetric technique. Figure 1 shows typical intrusion curves of two chars – one high and one low in porosity. The shape of each curve indicates that at low pressure the interparticle voids of the char are rapidly filled up by mercury and at high pressure the char is compressed. The combined macro- and mesopore volume was obtained from the difference between the intrusion volume at 413 kPa and the point at the intercept line (at 413 kPa) extrapolated from the high pressure curve. It should be noted that both the combined volume and the gradient of the intercept line, which reflect the compressibility of the char, are much greater for the Onakawana char (low rank lignite char) than those for the Canmore char (high rank semi-anthracite char). The gradual decrease in the combined volume and the gradient of the intercept line with increasing coal rank can be seen in Table 3.

The mercury density of the chars was measured at 413 kPa assuming that the intrusion volume at this pressure excluded the interparticle voids but included the intraparticle pores[10]. The combination of this apparent density and the 'true' density determined by the helium replacement method yielded the total pore volume from which the micropore volume could be calculated by difference. Open porosity defined as the percentage of total volume of open pores (cavities or channels communicating with the surface of the solids) relative to the char volume could also be calculated. The results are shown in Table 3 and graphically represented in Figure 2. It is seen that as the rank of the parent coals increases, both the open porosity and the 'He accessible' pore volume of the chars decrease. It is interesting to note that chars and their parent coals show the same trends in this respect[1] except that chars are much more porous but less compressible. Evidently, more open pores were generated during the devolatilization process with the low rank coals showing the most pronounced effect.

Figure 3 depicts sorption curves for the Sundance char using N_2 and CO_2 as adsorbates. The N_2 sorption curves have a characteristic feature of hysteresis loop which can be classified as Type IV isotherm for mesoporous solids[11]. In contrast, N_2 isotherms of coals appear to be of Type II which is characteristic of less porous solids[1]. The existence of a hysteresis loop in the sorption isotherm is explained by one theory as having 'ink bottle' pores[12] where capillary condensation and evaporation of the adsorbate take place at different relative pressures.

Both N_2 and CO_2 surface areas of char samples were calculated from adsorption curves. The results are shown in Table 3. It appears that a linear correlation exists between the CO_2 surface areas of the chars calculated from BET and DRK methods. Thus, only BET surface areas will be considered in this paper. The relationship between surface area and the rank of the parent coals is depicted in Figure 4. In general, the surface area decreases sharply as the coal rank increases until a certain value of C^{daf} is reached. In this study there seems to be a 'parabolic' type correlation between the N_2 and the CO_2 surface area (Figure 5) with the former always smaller than the latter. This phenomenon has been explained by Mahajan[13] that (1) CO_2 is a smaller molecule (3.3Å) than a N_2 molecule (3.64Å); (2) a CO_2 molecule has higher kinetic energy and thus higher diffusion rate at 195 K than the N_2 molecule at 77 K. This will permit the CO_2 to be more accessible than the N_2 to the micropores; (3) some pores at 77 K may shrink to such an extent that they are no longer accessible to N_2 molecules. Other interpretations can also be found in the literature that CO_2 adsorption may be influenced by the quadrupole moment of CO_2 molecule interacting with the oxygen functionalities present on the carbon surface[14] and that higher surface area may be caused by a CO_2-induced swelling effect[15].

The observation that CO_2 surface area is always larger than that of N_2 for chars applies also to the 12 parent coals of this study[16]. Comparison of a char with its parent coal shows that the char has much larger N_2 surface area until the % C^{daf} of parent coal reaches ∿78% (Figure 4). Beyond that, the N_2 surface areas of both char and coal are small and comparable[1]. The CO_2 surface area of a low rank coal is also smaller than that of its corresponding char. Both the surface areas of coal and char decrease with increasing C^{daf}. However, the CO_2 surface area of the coal rises again after a minimum is reached[16] but that of the char continues to

decline. Thus at high C^{daf}, the char has a lower CO_2 surface area than its parent coal. At present one may speculate that the polar interaction of CO_2 with O_2 groups and/or CO_2-induced swelling of coals may play an important role in causing the rise of the CO_2 curve. Also, CO_2 seems to diffuse at different rates into the pores of coals of different ranks. Nandi and Walker[17] estimated that for an arbitrary equilibration time of 30 min for each sorption point, the anthracite coal would have a much higher percentage of the pore volume filled with CO_2 at 195 K than a bituminous coal.

The fact that considerably good correlation exists between N_2 and CO_2 surface areas for chars (Figure 5) but not for coals[10] suggests that coals may contain some interfering components which are removed during the devolatilization period.

Pore volume can also be derived from CO_2 adsorption (using DR equation[6]) and N_2 desorption (using the BJH[3] and the Kelvin[4] equations). The resulting 'CO_2 accessible' micropore volume and 'N_2 accessible' pore volume are shown in Table 4 along with the 'He accessible' pore volume. It is apparent that for the same char the value obtained from the He technique is the greatest followed by those from the CO_2 and N_2 sorption methods in descending order. In this study the pore volume obtained from the N_2 desorption branch covers the widest diameter range (20 to 600Å) and yet its value is, in general, the smallest among the three for the same char sample. This suggests that many of the pores in chars have openings less than 20Å which are inaccessible to N_2 at 77 K but accessible to He (the smallest molecule among the three) at room temperature. Some of these small pores (<20Å) may have resulted from bigger pores due to pore shrinkage at very low temperature.

In spite of the discrepancy in magnitude, good correlation is obtained between He and CO_2 micropore volumes. This is shown in Figure 6 which indicates the existence of two linear regions. The first, with steeper slope, shows the greater difference between He and CO_2 micropore volumes of chars derived from the high rank coals. Figure 7 depicts the effect of He micropore volume on N_2 and CO_2 surface areas of 12 chars. As expected, surface areas increase with increasing micropore volume. However below 0.13 mL/g there is little or no change in the N_2 surface area. This is probably due to the temperature effect which is important in the gas sorption technique but not in the He pore volume measurement.

The relationship of reactivity of 8 Canadian coal-derived chars with their parent coal ranks has been reported previously[2]. The char reactivity is defined as the ratio of the reaction rate observed in the initial linear region in a mass conversion-time plot over the initial mass of the char on dry and ash-free basis. It is apparent from Figure 8 that the addition of four more chars does not change the linear relationship between the reactivity and the carbon content of parent coal in a semilog plot except the Mt.Klappan char has higher reactivity than expected. This anthracite char also shows abnormally high surface area and pore volume with respect to the C^{daf} of its coal.

The mechanism of char gasification under the conditions of this study has previously been explained using the shrinking core model[2]. The reaction occurs on the outside surface of the char particle but the reaction surface will move inwards as the gasification proceeds, leaving an ash layer behind without changing the external dimension of the char particle. Previous experimental data further confirmed that the initial stage of reaction is kinetically controlled by chemical reaction at the surface of the core and the later stage by the gas diffusion through the ash. It is thus expected that the pore structure of the char will have great impact on the char reactivity. Figure 9 indicates the relationship between the char reactivity and the surface areas measured by two gas adsorption techniques. In both cases, the char reactivity increases rapidly, then slows down gradually and finally rises sharply as the surface area increases. The fast increase at the final stage is attributed to the fact that the three lignite chars (Onakawana, Bienfait and Coronach) with high surface areas are comparatively rich in calcium (6.17%, 2.84% and 4.24% CaO respectively) which may induce catalytic gasification[2,9,18]. Walker[9] reported that the carboxyl groups in lignites decompose upon pyrolysis, liberating CO_2 and resulting in well dispersed metals or metal oxides which act as catalysts for char gasification. The most significant in-situ catalyst in chars produced from low rank coals is CaO due to its abundance and better dispersion during pyrolysis. In contrast, Ca in the higher rank coals is present primarily as larger particles of calcite. Other metals such as Mg, Na, K and Fe have also been reported as good catalysts but with some uncertainty[2,9,18].

The effect of He pore volume on char reactivity is shown in Figure 10. It can be seen that the char reactivity increases with both total and micropore volumes. Its relationship with the total pore volume is linear but that with the micropore volume deviates from linearity when the three lignite chars are included in the plot. The pronounced effect of micropore volume of the lignite chars may be due to the presence of more feeder pores (maro- and meso-pores) as indicated in Figure 2B. Walker [9] suggested that the differences in reactivity can be attributed to three main factors: (i) concentration of active sites (which usually exist in micropores); (ii) accessibility of the reactant gas to active sites (in this case it will require more feeder pores) and (iii) catalysis. It is noted that all these factors prevail for lignite chars.

In an earlier study[1] on physical properties of eleven Canadian coals a correlation has been established between helium density and the carbon content of the coals of different origins (Canadian, Japanese, American, British and Czechoslovakian coals). An attempt is made to determine whether the same correlation applies to the chars of this study. The helium densities of 12 coal-derived chars were then corrected to the mineral-matter-free basis using a density value of 2.7 g mL^{-1} for the mineral matter[10]. After correction, the data points of chars were plotted along with those of their parent coals (Figure 11). As expected, the helium density of the char, or coal, decreases initially with increasing carbon content, passes through a minimum and increases to reach a value of 2.25 for pure graphite[19].

CONCLUSION

It has been found that the open porosity, total pore volume, micropore volume, N_2 and CO_2 surface areas, gasification reactivity and the apparent compressibility of chars derived from 12 Canadian coals decrease with increase in the carbon content of the coals. Compared with their parent coals, chars are more porous (especially for lignite chars) but less compressible. Lignite chars are characterized by high concentrations of feeder pores, active sites and Ca which may act as a catalyst in gasification. The combination of these factors has greatly enhanced the reactivity of lignite chars. The char reactivity can be correlated with surface areas and pore

volumes determined by different methods. The previously found relationship between the helium density and the carbon content of the coals can be further extended to chars when the helium density is corrected for the mineral content of the char.

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Table 1 - Chemical analyses of coal and char samples

Sample (rank)	Proximate analysis (wt %)			Ultimate analysis (wt %)				
	Ash	Volatile matter	Fixed carbon[a]	C	H	S	N	O[a]
Onakawana (lignite)								
coal	26.3	39.6	34.0	51.7	3.5	1.8	0.7	15.9
char	30.7	6.9	62.4	67.8	0.6	1.2	1.0	-
Bienfait (lignite)								
coal	10.9	44.1	45.0	62.8	4.3	1.8	1.2	19.1
char	15.5	6.0	78.5	81.6	0.6	0.7	1.5	0.1
Tulameen (subbituminous)								
coal	15.2	32.4	52.4	64.2	4.2	4.2	1.5	10.7
char	23.9	2.5	73.6	73.4	0.7	0.5	1.4	0.2
Mt. Klappan (anthracite)								
coal	21.6	9.1	69.4	69.4	2.1	0.4	1.0	5.5
char	24.5	1.6	74.0	73.4	0.7	0.4	1.1	-

[a] Determined by difference

Table 2 - Chemical analyses of ashes in char samples (wt %)

Char Sample	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	P_2O_5	CaO	MgO	SO_3	Na_2O	K_2O
Onakawana	11.8	3.71	2.21	0.26	0.09	6.17	1.68	2.99	0.39	0.25
Bienfait	3.89	2.36	1.26	0.15	0.21	2.84	0.56	1.80	1.72	0.05
Tulameen	16.1	3.30	2.10	0.20	0.06	0.19	0.18	0.04	0.05	0.46
Mt. Klappan	16.0	4.75	1.30	0.33	0.13	0.28	0.40	0.02	0.37	0.28

Table 3 - Carbon content and physical properties of chars

Char Sample	C^{daf} (wt %) Parent coal	Char	Helium density (g mL^{-1})	Mercury density (g mL^{-1})	Open porosity (vol %)	Helium accessible pore volume (mL g^{-1}) Total	Macro- and meso-pore	Micro-pore	Gradient x 10^5 (mL g^{-1} MPa^{-1})	Surface area (m^{2}g^{-1}) N$_2$ BET	CO$_2$ BET	CO$_2$ DRK
Onakawana	70.2	97.9	2.130	0.993	53.4	0.537	0.274	0.263	16.6	134	279	399
Bienfait	70.5	96.6	2.020	1.293	36.0	0.278	0.093	0.185	16.8	38.2	246	326
Coronach	70.6	97.8	2.089	1.354	35.2	0.260	0.066	0.194	10.0	79.9	241	348
Tulameen	75.7	96.4	1.937	1.048	45.9	0.438	0.267	0.171	13.9	41.1	212	287
Sundance	75.7	99.4	2.177	1.491	31.5	0.211	0.051	0.160	11.6	81.5	184	230
Prince	78.1	94.2	2.060	1.493	27.6	0.185	0.112	0.073	10.5	4.01	20.6	23.7
Coalspur	79.5	98.3	2.057	1.510	26.6	0.176	0.062	0.114	5.1	1.89	46.5	67.1
Devco	87.1	97.0	1.913	1.749	8.6	0.049	0.040	0.009	4.7	1.16	5.91	6.42
Mt. Klappan	88.5	97.1	2.096	1.420	32.3	0.227	0.088	0.139	11.5	6.73	121	176
Balmer	89.5	97.6	1.936	1.626	16.0	0.099	0.071	0.027	4.3	1.12	5.35	6.68
McIntyre	90.5	98.1	2.023	1.562	22.8	0.146	0.099	0.047	3.9	2.17	10.5	15.6
Canmore	92.0	97.8	1.761	1.632	7.3	0.045	0.030	0.015	6.3	0.95	5.58	5.71

Table 4 – Comparison of micropore volumes
obtained by different methods

Char Sample	Micropore volume (mLg^{-1})		N_2 accessible pore volume (mL g^{-1})
	Helium accessible	CO_2 Accessible	
Onakawana	0.263	0.136	0.108
Bienfait	0.185	0.111	0.043
Coronach	0.194	0.119	0.059
Tulameen	0.171	0.098	0.045
Sundance	0.160	0.078	0.077
Prince	0.073	0.008	0.015
Coalspur	0.114	0.023	0.008
Devco	0.009	0.002	0.009
Mt. Klappan	0.139	0.060	0.026
Balmer	0.027	0.002	0.008
McIntyre	0.047	0.005	0.014
Canmore	0.015	0.002	0.009
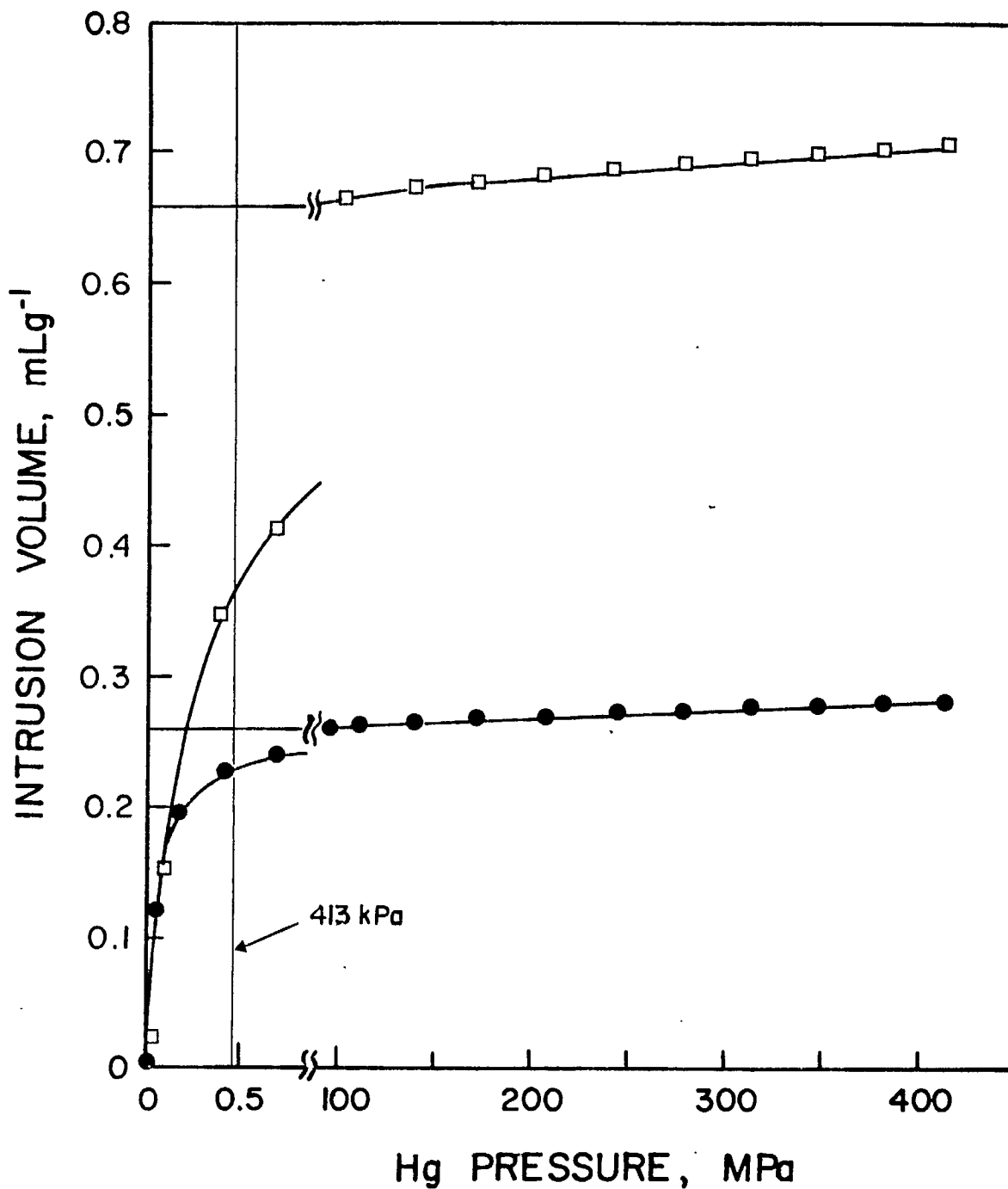
Figure 1 Mercury intrusion curves of coal-derived chars.
□, Onakawana char; ●, Canmore char
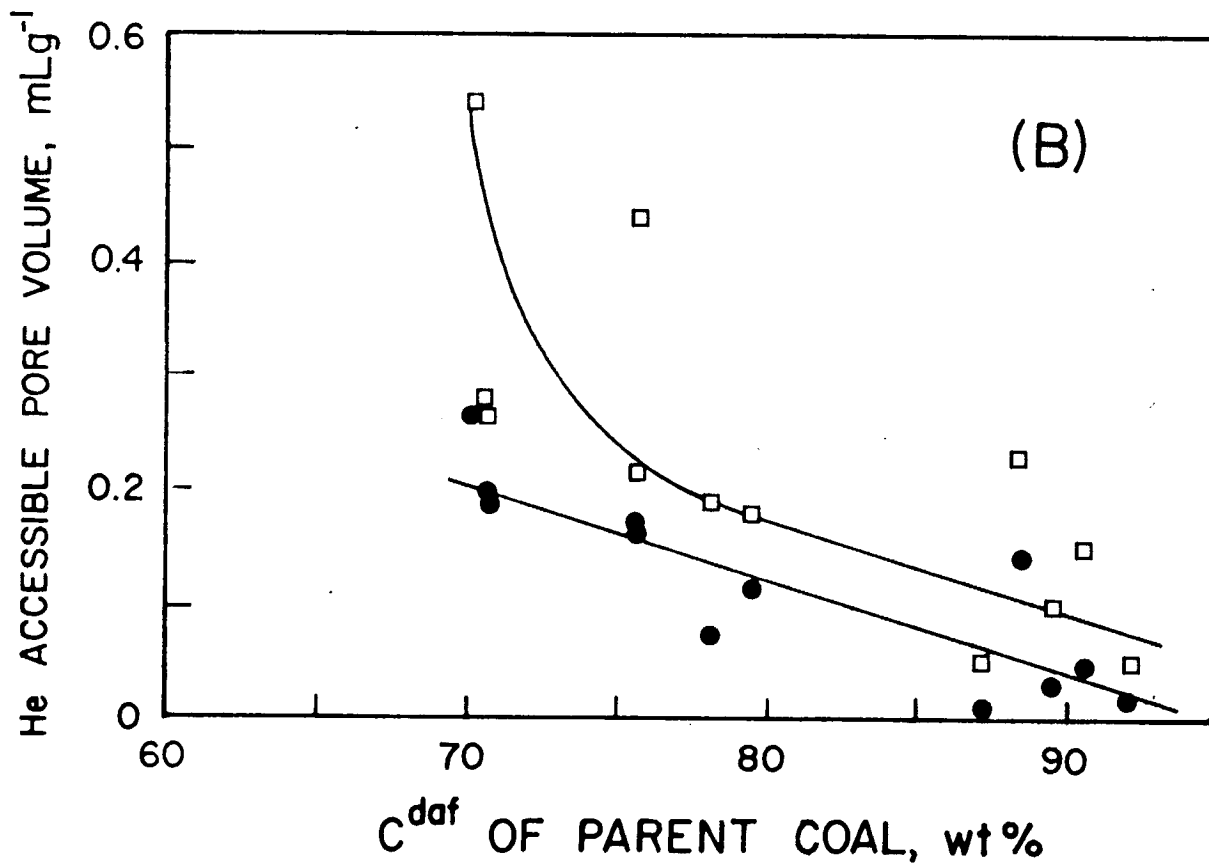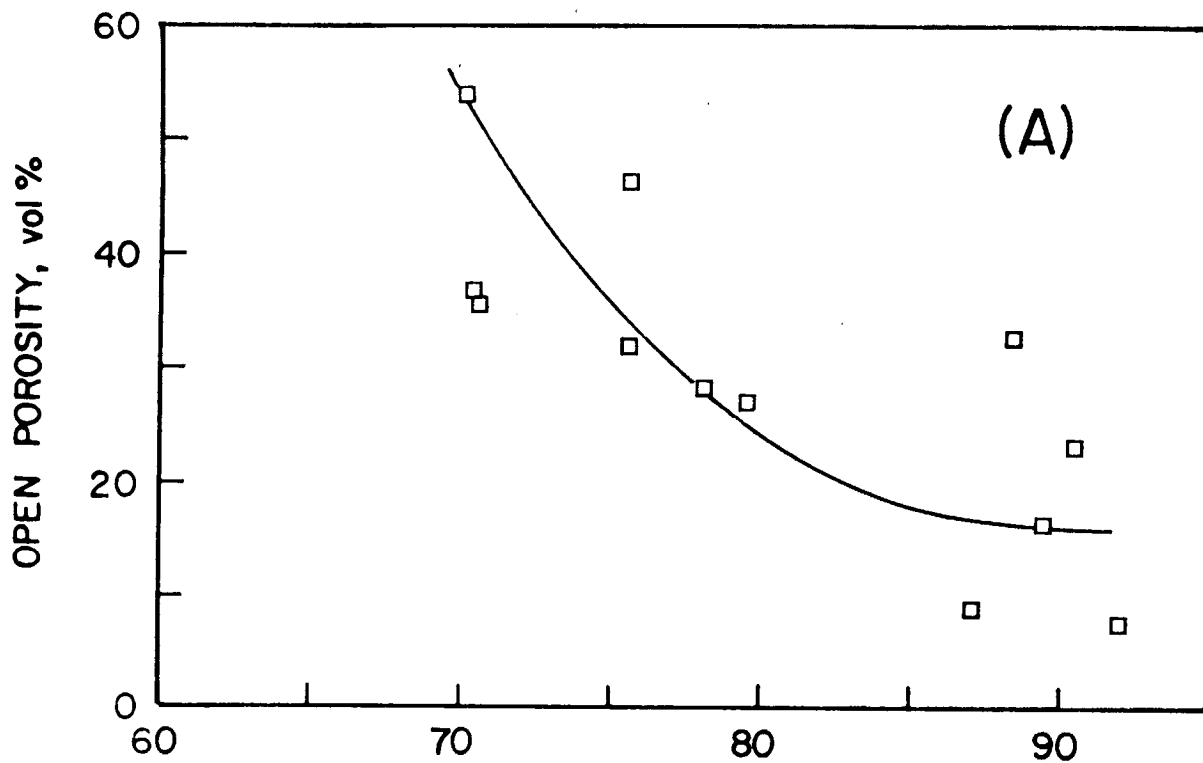
Figure 2 Variation of open porosity (A) and pore volume (B) with carbon content of coal-derived char. In (B): □,total and ●,micropore volume
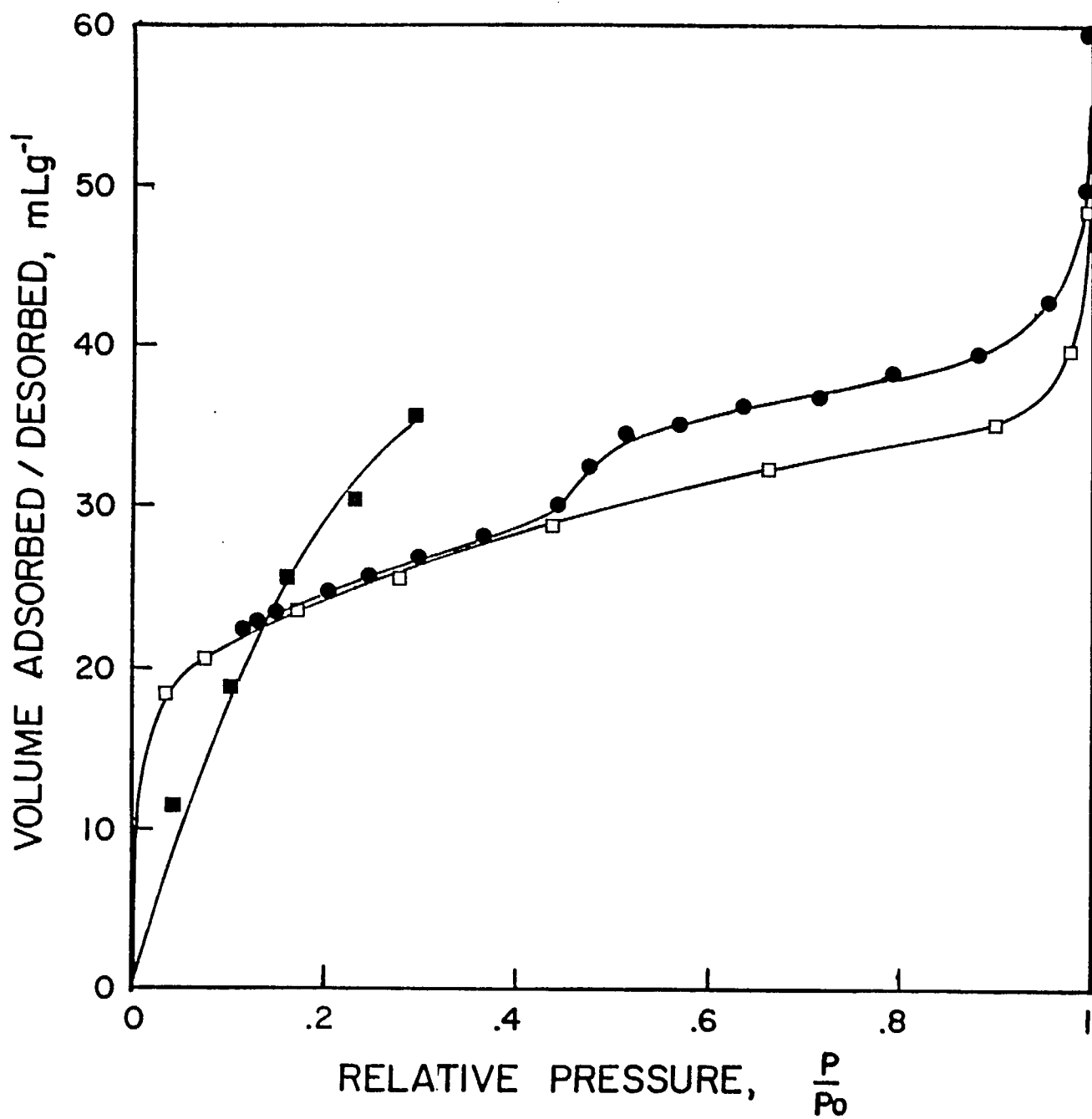
Figure 3 Gas sorption isotherms of Sundance char. ☐ , N_2 adsorption and
● , N_2 desorption at 77 K; ■ CO_2 adsorption at 195 K
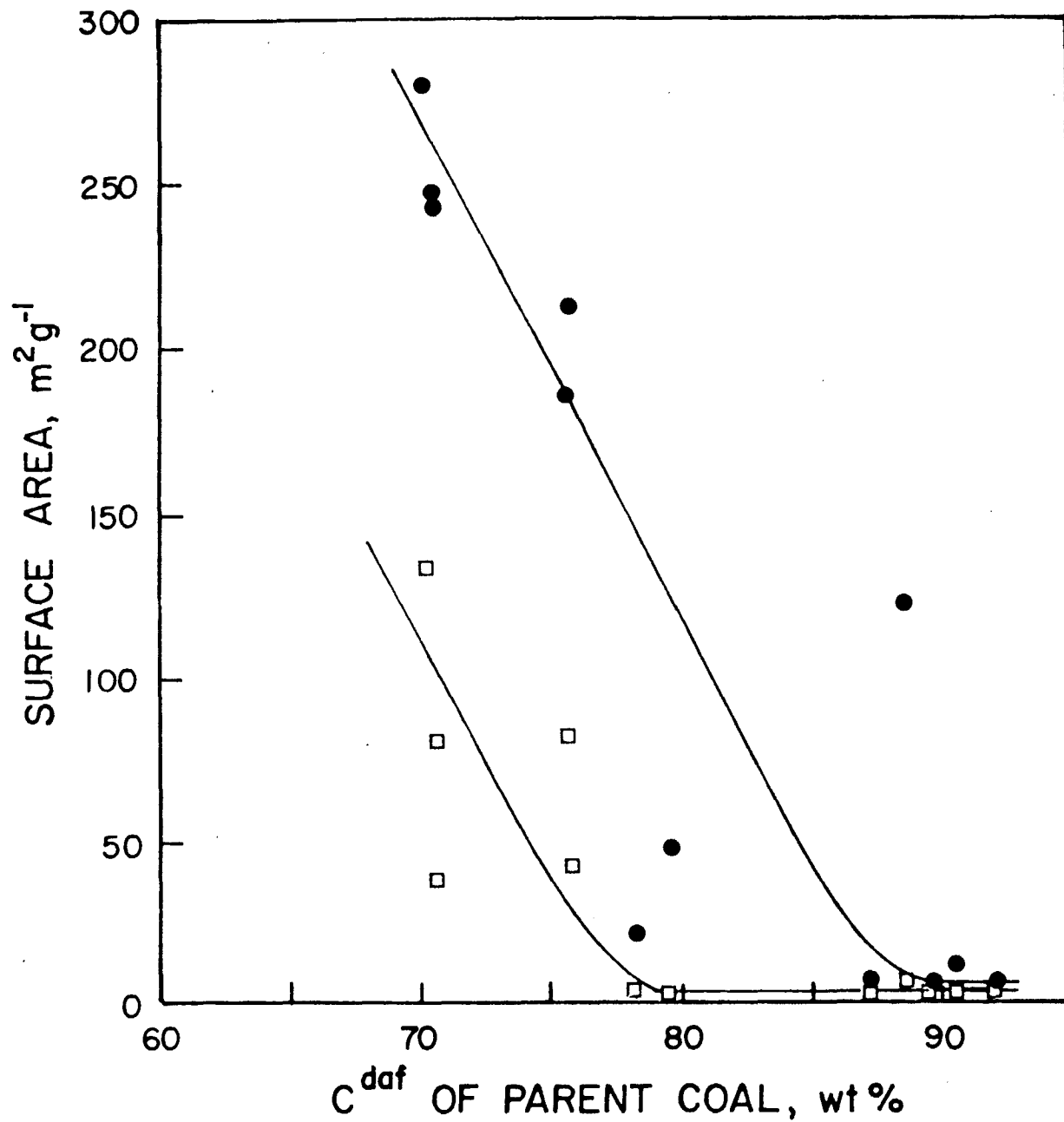
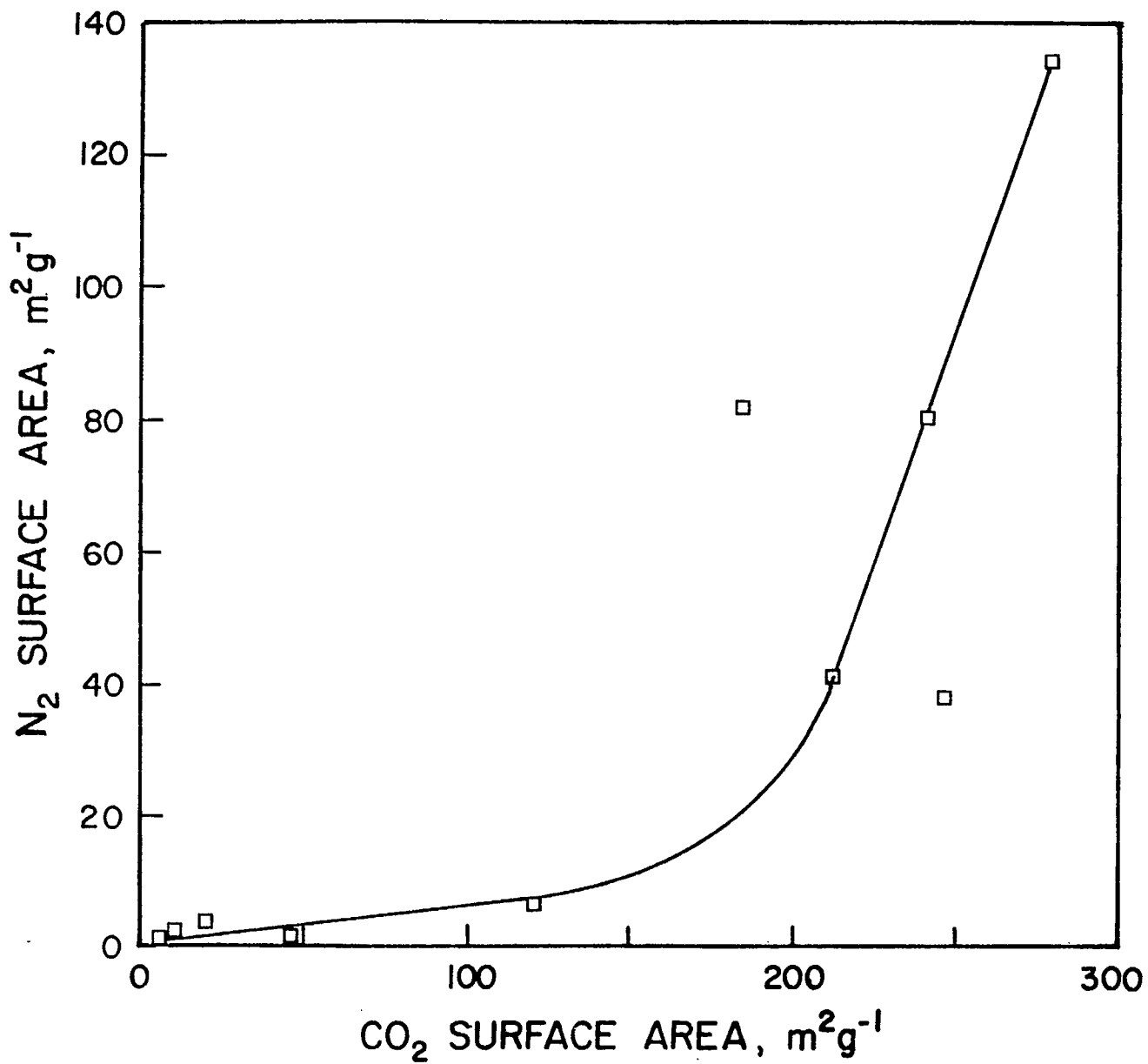Figure 4 Relationship between surface area and carbon content of coal-derived char. □ , N_2 and ●, CO_2 surface area

Figure 5 Correlation between N_2 surface area and CO_2 surface area of coal-derived char
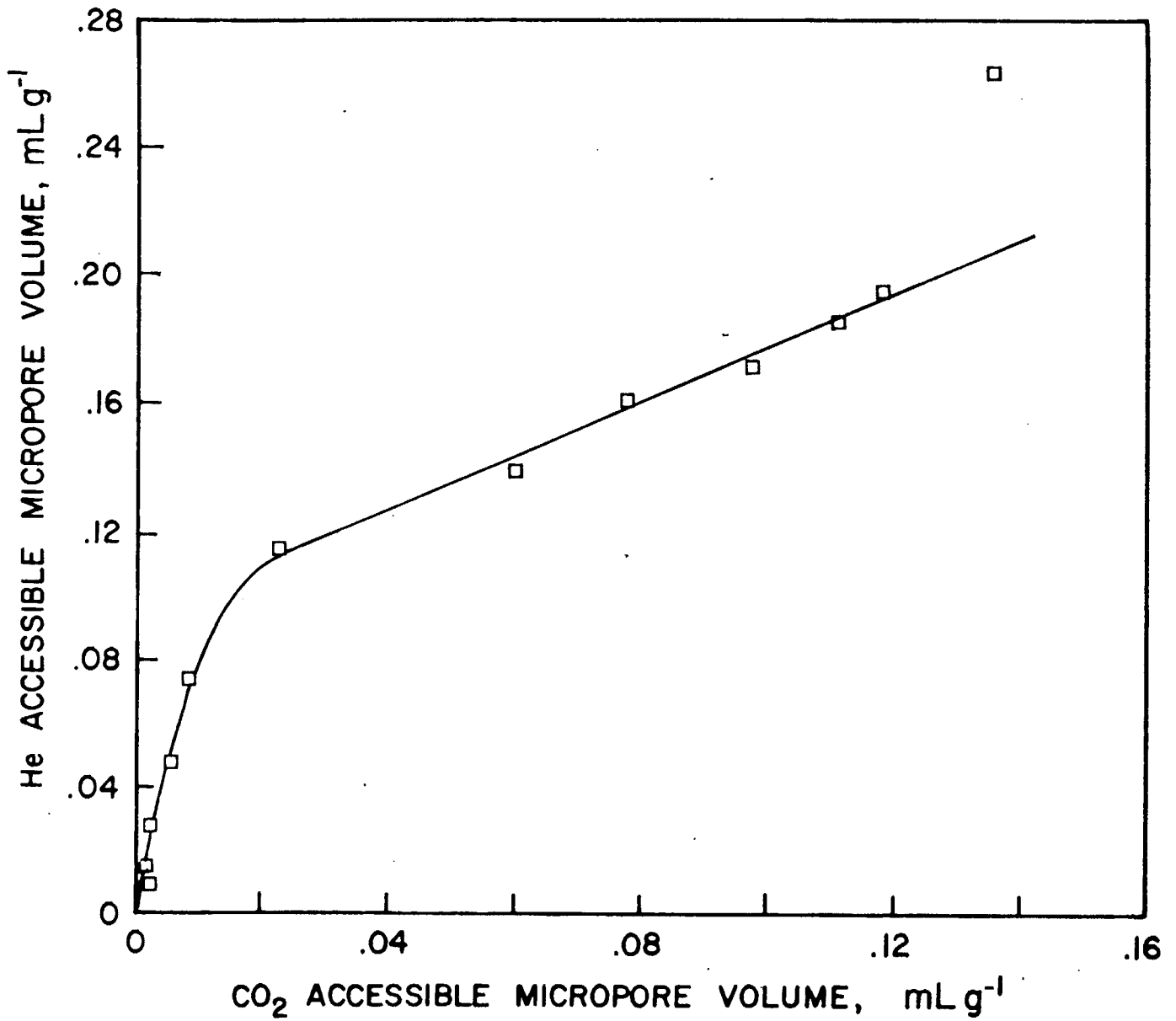
Figure 6 Correlation of He accessible micropore volume with CO_2 accessible micropore volume of coal-derived char
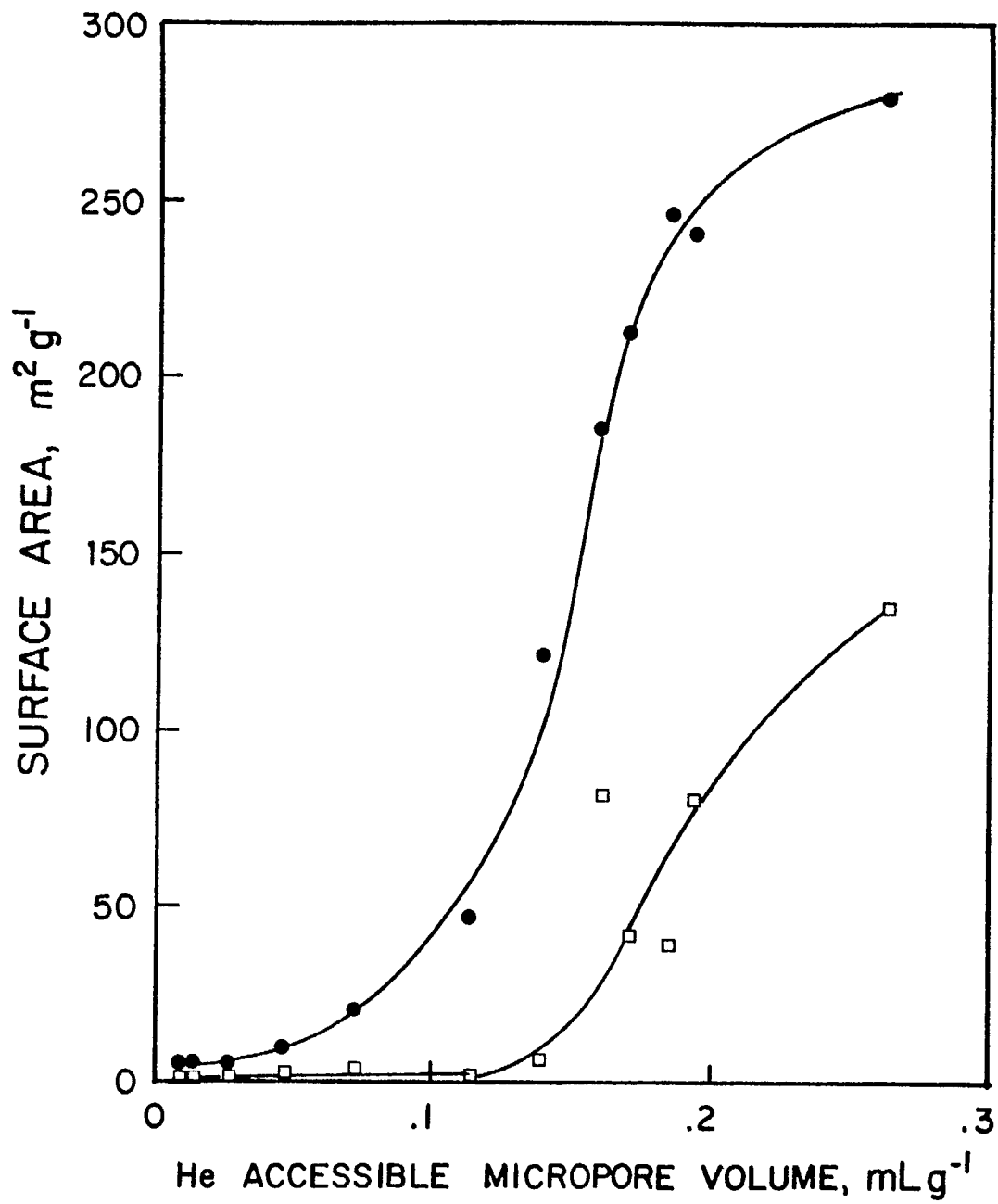
Figure 7 Effect of He micropore volume on surface area of coal–derived char. □ , N_2 and ●, CO_2 surface area
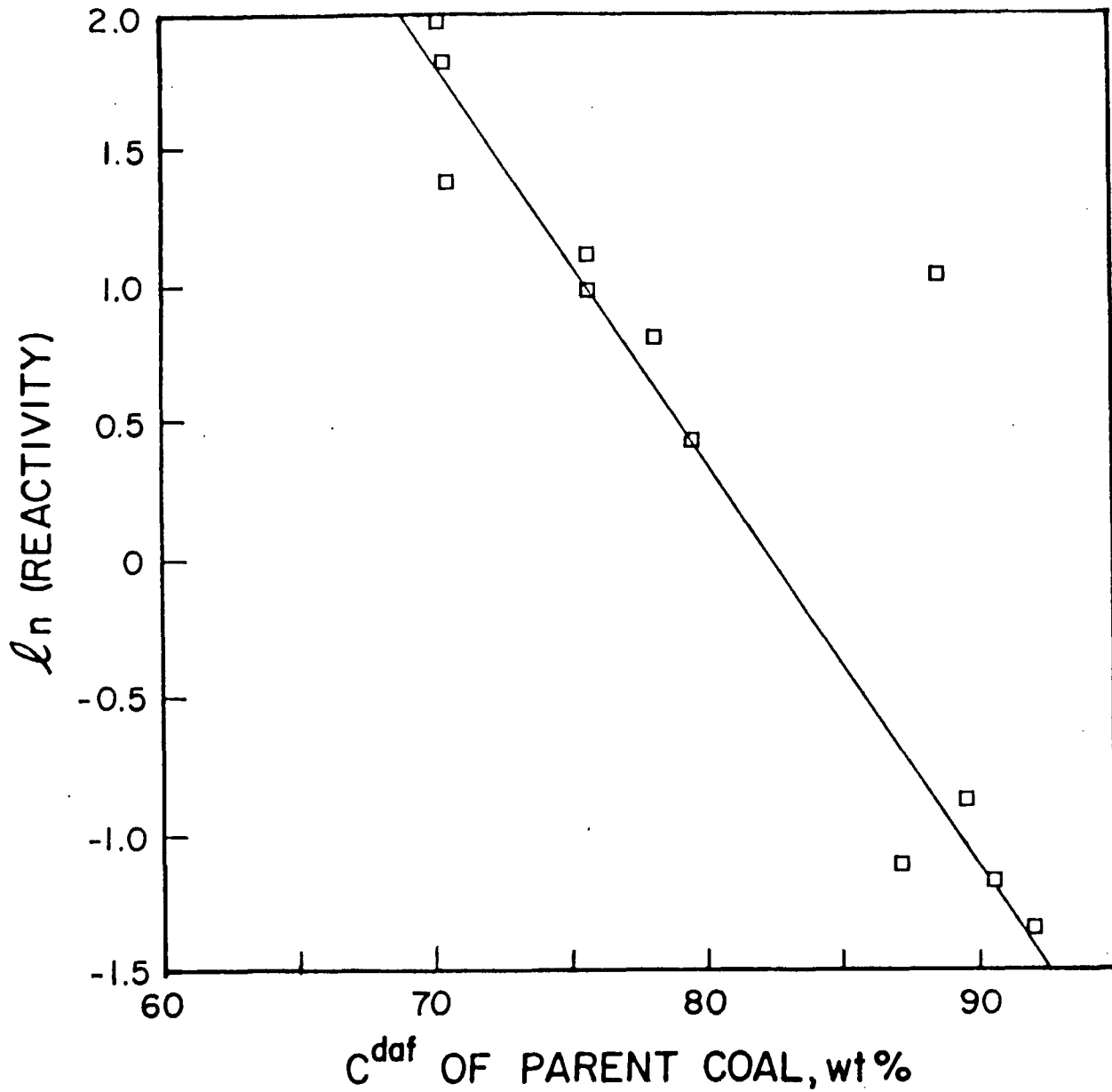
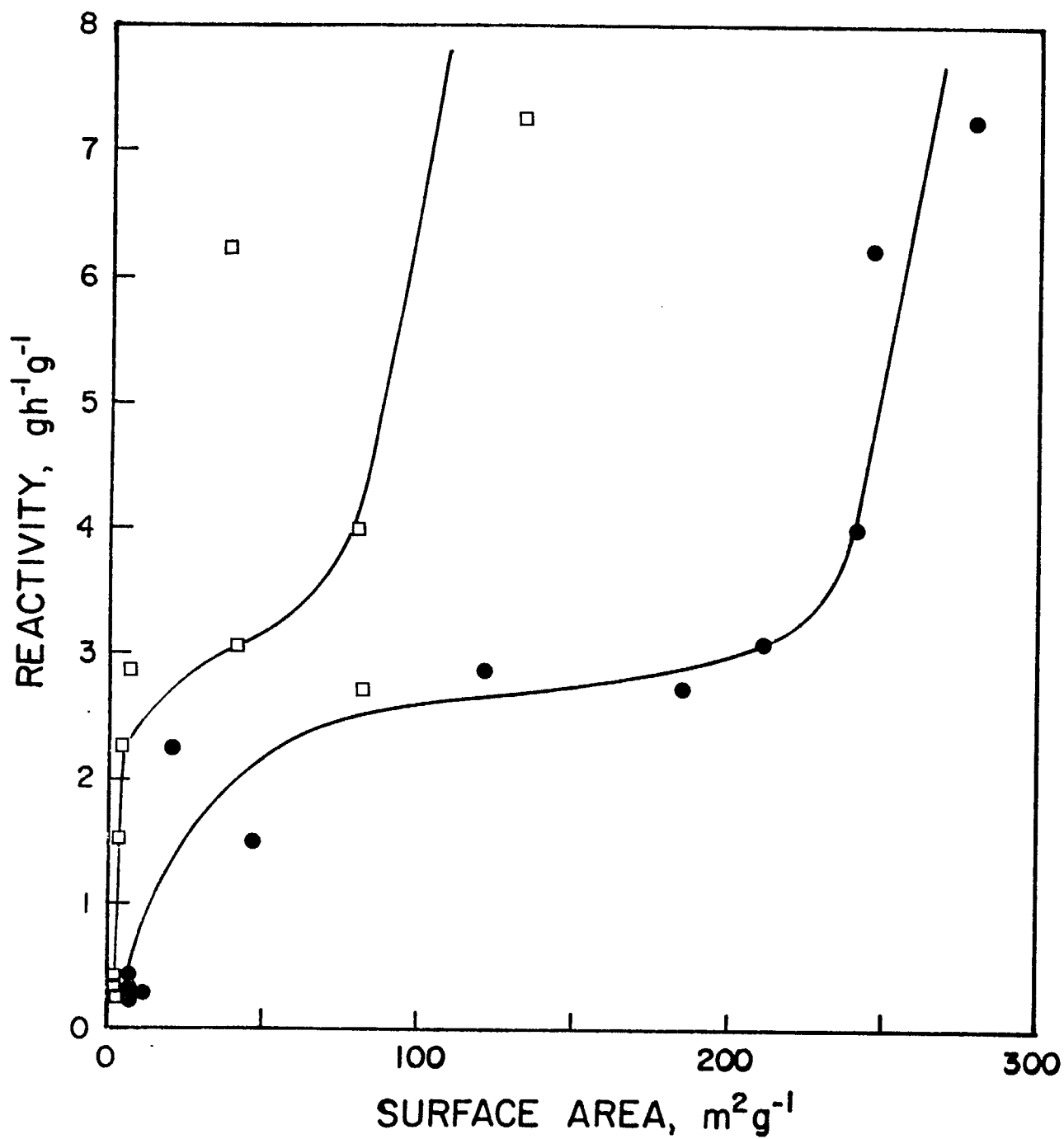Figure 8 Dependence of char reactivity on the carbon content of the parent coal

Figure 9 Correlation between reactivity and surface area of coal-derived chars. □, N_2 and ●, CO_2 surface area
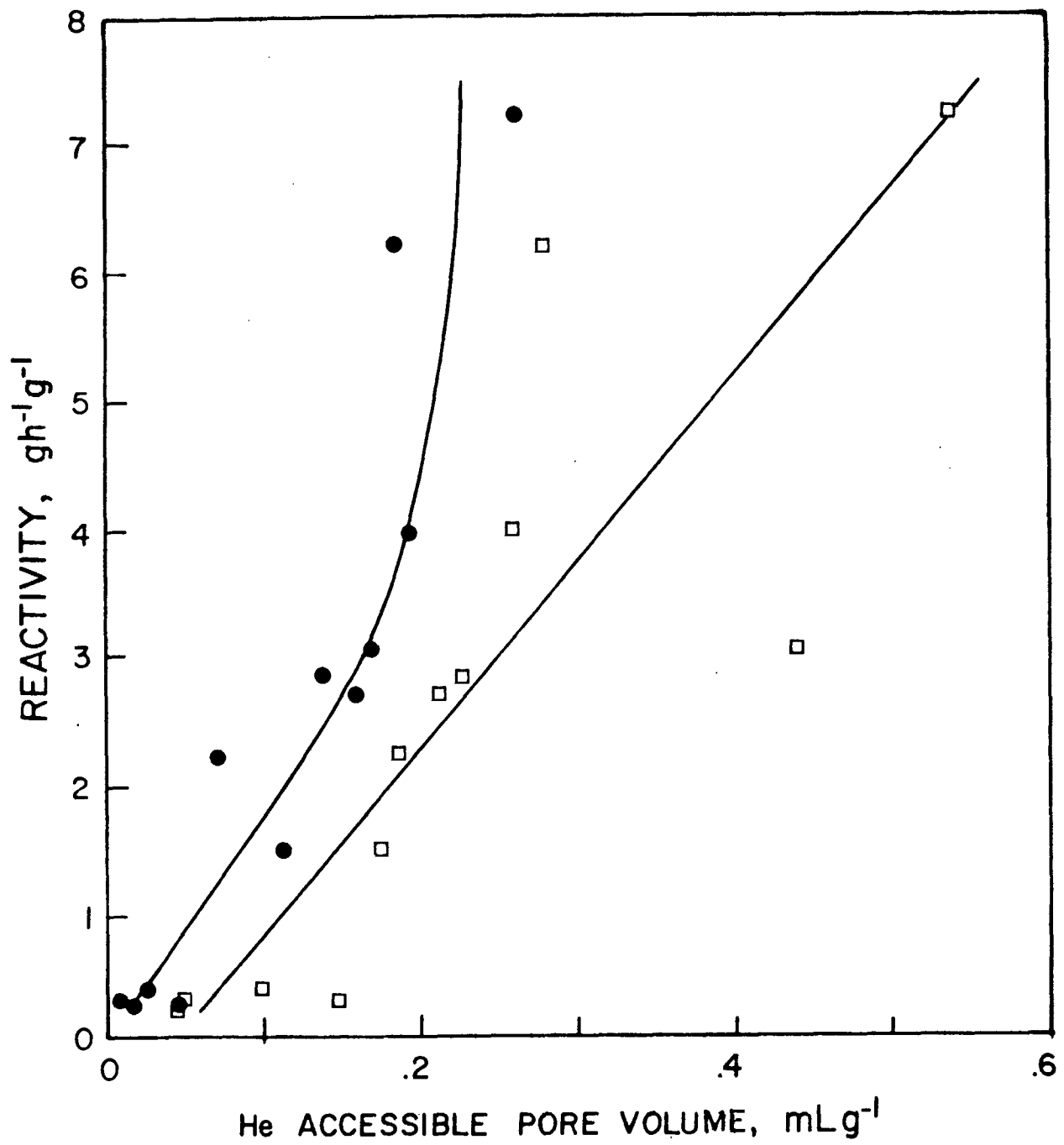
Figure 10 Relationship between reactivity and He pore volume of coal-derived char. □ , total and ●, micropore volume
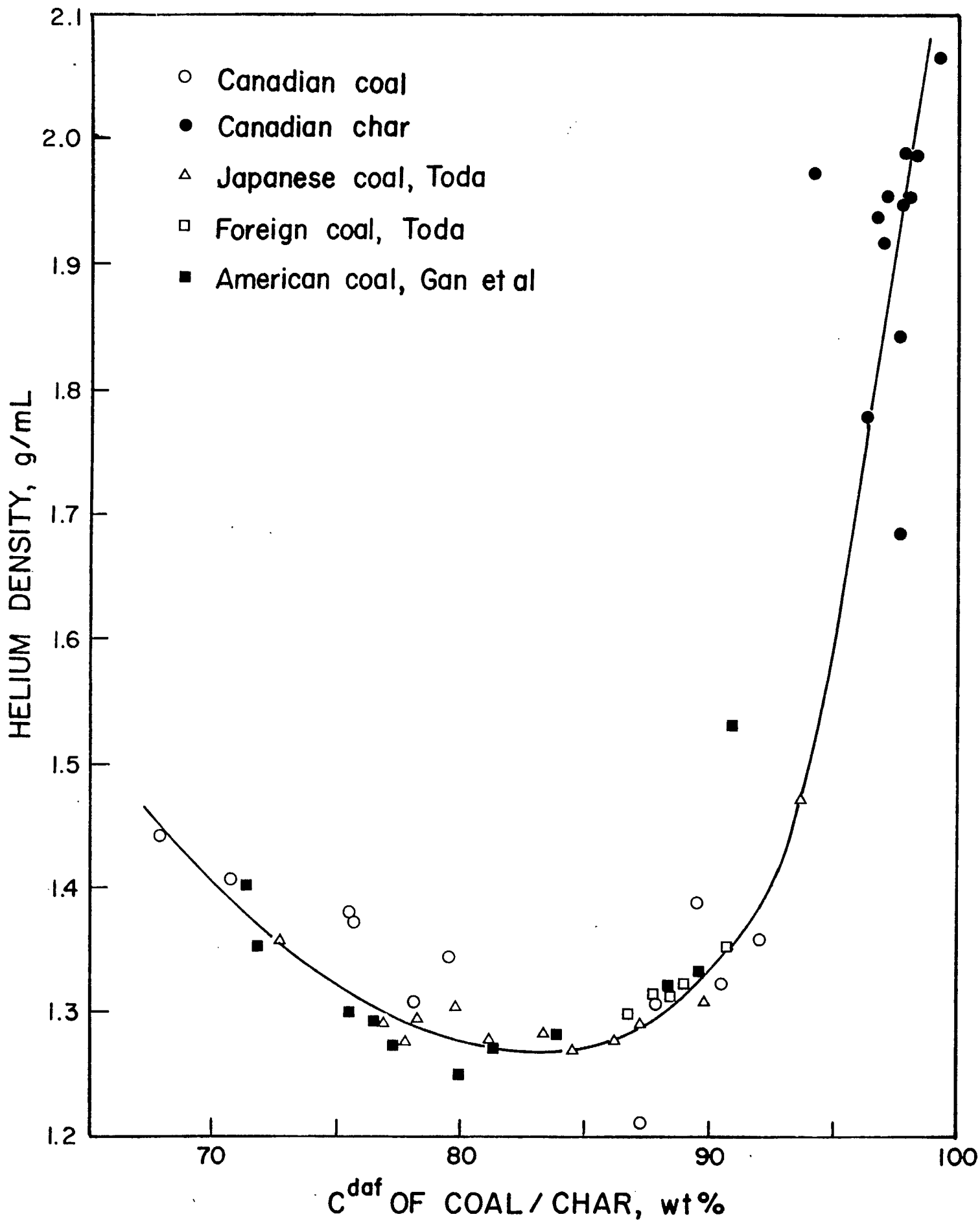
Figure 11 Relationship between helium density (mineral matter free) and carbon content of coal/coal-derived char