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SOME PHYSICAL PROPERTIES OF CANADIAN COALS
AND THEIR EFFECTS ON COAL REACTIVITY

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THEIR EFFECTS ON COAL REACTIVITY

by

Siauw H. Ng*, D.P.C. Fung* and S.D. Kim**

ABSTRACT

The pore volume, surface area and compressibility of eleven Canadian coals, varying in rank from lignite to semianthracite, have been determined by mercury porosimetry, gas adsorption method and relations derived from helium and mercury densities. The total pore volume was measured in a diameter range of 0.2 nm - 2.98 μm which was subdivided into two groups, namely the micropore region ($<0.0036 \mu\text{m}$) and the combined meso- and macropore region (0.0036 - 2.98 μm). It has been determined that the porosity of the eleven coals studied varies from 2 to 39%.

It has been found that the total pore volume, micropore volume, surface area and the apparent compressibility of these coals decrease with increase in the carbon content, or the rank of the coals. The effect of the total pore volume, micropore volume and surface area on chemical reactivity of the coal is discussed separately. A good correlation is obtained between the carbon content and helium density of the coal after correction is made for the mineral content.

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QUELQUES PROPRIETES PHYSIQUES DE CHARBONS CANADIENS
ET LEURS EFFETS SUR LA REACTIVITE DU CHARBON

par

Siauw H. Ng*, D.P.C. Fung* et S.D. Kim**

RESUME

Le volume des pores, l'aire de la surface et la compressibilité de onze charbons canadiens, allant du lignite au semi-anthracite, ont été déterminés par le biais de la porosimétrie au mercure, de l'adsorption gazeuse et des relations établies à partir des densités mesurées à l'aide de l'hélium et du mercure. Le volume total des pores a été mesuré pour une tranche de diamètres variant de 0,2 nm à 2,98 μm qui a été divisée en deux groupes: les micropores ($< 0,0036 \mu\text{m}$) et les mésopores et macropores combinés (de 0,0036 à 2,98 μm). On a déterminé que la porosité des onze charbons étudiés varie de 2 à 39%.

On s'est rendu compte que le volume total des pores, le volume des micropores, l'aire de la surface et la compressibilité apparente diminuent à mesure qu'augmente la teneur en carbone ou varient selon le rang de charbon. Les effets du volume total des pores, du volume des micropores et de l'aire de la surface sur la réactivité chimique du charbon font l'objet d'une section distincte. On peut identifier une nette interrelation entre la teneur en carbone du charbon et sa densité mesurée à l'aide de l'hélium après que la correction relative à la teneur en minéraux est effectuée.

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INTRODUCTION

During the last few years, we have studied the effect of chemical properties on the gasification reactivity of various Canadian coals as part of a national R & D program on coal utilization in Canada¹⁻⁴. To continue this effort, we also examined the physical properties of these coals with the emphasis on their pore system. In the past decade, several investigations⁵⁻¹⁰ were conducted in this area because of the marked influence of pore structure on the rate of interaction of coal with gases and liquids during gasification and combustion of coals, coal drying, coal flotation and treatment of waste water through a coal filtration bed. Eleven coals, most of which had been used in our previous reactivity studies, were selected for pore structure measurement. We used a combination of mercury porosimetry and helium density measurement to determine the pore volumes in the combined macro-meso and in the micro ranges. The surface area (mainly contributed by micropores) was measured by the nitrogen adsorption technique.

EXPERIMENTAL

Materials

After pulverization, the coal sample (particle size 425 μm - 212 μm) was pre-dried in an oven at 378°K for 1 hr. The chemical analyses of these samples are shown in Table 1.

Triply distilled mercury was used for the porosimetric study. Nitrogen and helium used for the gas adsorption and density measurement were of high purity grade.

Apparatus

Mercury densities and pore volumes in the range of macropore (2.98 - 0.06 μm) and mesopore (0.06 - 0.0036 μm) were determined using a commercial computerized high pressure mercury porosimeter (Micromeritics AutoPore 9200) with a maximum operational pressure of 413 MPa. This pressure corresponds to a pore diameter of 0.0030 μm according to the Young and Laplace equation¹¹.

$$d = \frac{-4\sigma\cos\theta}{p}$$

where σ is the surface tension of mercury, θ the contact angle and P the applied pressure. Substitution of the values of σ (0.485 Nm^{-1}) and θ (130°)¹² in this equation gives

$$d = \frac{1.23}{P}$$

when d is expressed in μm and P in MPa. The mercury penetration technique is based on the behavior of a nonwetting liquid (mercury) in capillaries, assuming that pores can be represented as cylinders.

Prior to each measurement, the sample (0.5 to 2.0 g depending on the size of dilatometer used) was outgassed under vacuum ($<3 \text{ Pa}$) before mercury was introduced into the dilatometer. Equilibration time for each pressure was 3 min. Prolonged equilibration time up to 10 min did not change the results significantly. The AutoPore 9200 is capable of making self-correction for the compression of glass dilatometer and mercury¹³.

The gas adsorption study and the helium density measurement were performed in a commercial unit (Micromeritics AccuSorb 2100E) applying the volumetric principle with dead-space correction. Samples were degassed under vacuum ($<10^{-3} \text{ Pa}$) at 373°K for 15h prior to measurement. Nitrogen surface area was determined at 77°K . Repeatability of surface area data was $\pm 5\%$. Helium density was determined by direct pressure measurement at both 77°K and room temperature.

For surface area measurement the equilibration time was 1 h for the first pressure point and 30 to 45 min for the rest. Equilibrium pressure (P_0) was gradually increased until it reached 30% or more of the saturation pressure (P_S). The BET equation¹⁴ was used for the calculation of the surface area assuming a value of 101.2 kPa for the P_S of nitrogen at 77°K and a value of 0.162 nm^2 for the cross section area of the nitrogen molecule¹⁵.

Open porosities and total pore volumes of the coal samples were calculated from their helium density and mercury density at 413 kPa.

RESULTS AND DISCUSSION

Recently, numerous studies^{1-4, 16-26}, have been conducted relating coal and char gasification rates to their chemical and/or physical properties. It has been found that in general the gasification reactivity of both coal and char in different atmospheres (O_2 , H_2 , H_2O , CO_2) decreases with

increase in coal rank^{2-4, 16, 17, 20, 22, 25, 26}. Walker Jr.²⁰ suggested that carbon gasification rates were promoted by three main factors which are (1) concentration of active carbon sites (2) accessibility of the sites to the reactive gas and (3) catalysis of gasification by mineral matter in the coals. The accessibility of the active sites and the catalytic effects of mineral matter will be discussed in relation to pore volume, surface area and chemical properties of coals.

Effect of pore volume on coal reactivity

Figure 1 shows a mercury intrusion curve for Sundance coal. In the region below 413 kPa, the rapid rise in the volume is caused by mercury filling the interparticle voids. It has been found that⁹ at 413 kPa all the interparticle voids would be filled for samples with particle sizes in the range of 425-212 μm and any further mercury penetration at higher pressure would be due to the presence of pores. The linear increase in the intrusion volume in the high pressure range was attributed²⁷ to compression of the coal. The gradient of the straight line (over the pressure range 20-345 MPa) in Figure 1 represents the apparent compressibility of the sample. The difference between the intercept of this straight line at 413 kPa and the experimental intrusion volume is the combined volume of the macro and mesopores^{7, 10} in the range of 2.98 - 0.0036 μm diameter. Because of interference⁷ from coal compression at high pressure, no attempt was made to separate the mesopore from the macropore volume.

The mercury density can be obtained from the Hg porosimetric study using known sample weight and amount of mercury in the fixed volume penetrometer. The results of mercury density at 413 kPa, combined macro and mesopore volume, and the gradient from the plot of intrusion volume versus pressure are listed in Table 2. In general, the gradient decreases as the rank of coal increases. The magnitude of the gradients for the 11 coals studied are in the same order as those reported^{7, 10}.

The total pore volume can be obtained from the difference between the reciprocals of mercury density and helium density (to be discussed later) assuming these two densities represent bulk (void free but pore included) and true (both void and pore free) densities. The results are shown in Table 2 and

Figure 2(A) for open porosity as a function of C^{daf} (dry-ash-free carbon content) of coal. It can be seen that the low rank coals are much more porous than the high rank coals and the dependence of open porosity on coal rank becomes smaller at C^{daf} above 80 wt%. The micropore volume of the coal samples decreases linearly with increase in the coal rank (Figure 2B). However, the majority of the pores of lignites are in the macro and mesopore ranges. This agrees with the results for the American coals⁹.

The relationship between the gasification rate and total pore volume of the coal is shown in Figure 3. For this study, data of gasification reactivity, which was based on the maximum rate of carbon conversion, were taken from our previous work² in which nine Canadian coals were gasified at 800-860°C in a laboratory fixed-bed reactor in an oxygen-enriched atmosphere, with or without steam present. From Figure 3, it can be seen that the reactivity of the coals remains relatively constant at total pore volumes below 0.070 mlg⁻¹, above which the reactivity rises rapidly. The turning point occurs at about 80 wt% carbon content where the micropore volume predominates over the meso and macropore volumes (Figure 2(B)). Jenkins et al²⁶ studied the reactivity of 21 heated coals and reported also that coals of more than 80 wt% carbon content produced chars or cokes of low reactivity. It is thus believed that both the big and small pores are important in determining the reactivity of coals. The "feeder" pores (macro and mesopores) provide the channels for the reactive gas to reach the active carbon sites in the micropores where the gasification reaction can take place.

It is interesting to note that the pore volume affects to the same degree the reactivity with different reacting gas mixtures whether or not there is steam present in the mixture. This is probably because under the condition used in the steam reaction system, the water gas shift reaction ($CO + H_2O \rightleftharpoons CO_2 + H_2$) is favoured over the carbon-steam reaction ($C + H_2O \rightleftharpoons H_2 + CO$)².

Effect of Surface Area on Coal Reactivity

Figure 4 represents a N_2 adsorption isotherm for Coronach lignite at 77°K. It can be classified as Type II isotherm for less porous solids according to BET classification¹⁴. Along the low pressure branch, monolayer adsorption is assumed to take place on the walls of the pores. Upon completion of mono-

layer adsorption, multilayer is gradually built up in the mesopores. The steep increase in adsorption at high relative pressure is due to the condensation of gas to bulk liquid on the macropores or on the surface of the solids. The surface area obtained from the N_2 isotherm at $77^\circ K$ using the BET equation represents the sum of the external area of the particles and the internal area contained in pores with diameters greater than 0.5 nm^9 . Figure 5 shows a correlation between the N_2 surface area and the carbon content, or the rank, of the coal. It has been found that the lowest rank coal, Onakawana lignite, has the highest surface area. The surface area curve declines as the coal rank increases and it levels off for coals with carbon content higher than 80 wt%.

Figure 6 shows that the coal reactivity² decreases with decrease in surface area which reflects primarily the size and volume of the micropores in coals. In the neighbourhood of $0.85 \text{ m}^2\text{g}^{-1}$ corresponding to 80 wt% carbon content (Figure 5), the reactivity drops drastically signifying that the small surface area is no longer a controlling factor. Other factors which may promote the reactivity of the lignites are their high contents of CaO , MgO and oxygen³. The alkaline mineral matter may act as a catalyst as gasification proceeds^{3,20}. Oxygen may be present in the form of cross-links in the coal structure, producing solids with thermal stability against softening upon heat treatment. Thus the solids have more open pores which would increase the reactivity²⁰. All these factors should be treated as a whole as the enhanced reactivity is due to their combined effects. The devolatilisation stage may be critical in deciding the reactivity in the subsequent gasification steps. In this connection, the amount and the nature of volatile matter may play an important role in determining the pore structure of the char produced after devolatilisation.

Contrary to the lignites, the higher rank coals have less active carbon sites, lower concentration of "feeder" pores, less alkali and alkaline mineral matter and less oxygen. Upon heat treatment, softening and caking problems may arise¹⁸ resulting in loss of much surface area and active site concentration. This explains the lower reactivities of higher rank coals when compared to lower rank coals.

Helium density as a function of coal rank

Helium density was treated in this study as true density to derive total pore volume of a coal in conjunction with the use of mercury density of the

coal. Helium is chosen as a fluid medium in displacement method because it is inert and is normally not adsorbed on the coal surface²⁸. Furthermore, with a diameter of 0.2 nm, helium is accessible to the smallest pores in coals²⁹ (0.5 to 1 nm in diameter).

Helium density was primarily determined at 77°K to make dead-space correction for the BET surface area measurement at the same temperature. However, it was observed in the subsequent study¹⁶ that a considerable amount of helium was adsorbed at liquid nitrogen temperature on the chars prepared from Coronach and Sundance coals. This phenomenon was also observed by other investigators³⁰⁻³². A previous study³² carried out at room temperature indicated that the adsorption of helium by vitrains was very small. Therefore, the helium density was also measured at room temperature. The results indicate that the temperature effect is negligible for the 11 coals studied.

Table 2 presents the helium density on a mineral matter containing basis. Both the high rank and low rank coals have higher helium density values than those from the intermediate rank coals. An attempt was made to compare our results with those reported by others^{9,33} for the American and Japanese coals. In general, they all show a parabolic pattern in the density-coal rank plot, but the data are somewhat scattered for the same coal rank. In most cases the Canadian coals have relatively higher helium density values presumably due to the higher mineral matter content. All the helium densities were then corrected to the mineral matter free basis using a density value of 2.7 gml⁻¹ for the mineral matter⁹. With the correction, a better correlation was obtained between the helium density and coal rank for all the coals regardless of their origins (Figure 7). The helium density of the coal initially decreases with increase in the carbon content, passes through a minimum at about 85 wt% carbon, and then increases again to reach a value of 2.25 for pure graphite³⁴. This parabolic trend was also observed on British^{35,36} and Czechoslovakian³⁷ coals. One may conclude that this trend is a general property of coals. This nature is presumably due to gradual changes in oxygen (atomic wt. 16) and hydrogen (atomic wt. 1) contents in coals as the carbon (atomic wt. 12) content increases.

Compressibility as a function of coal rank

The compressibility of 11 Canadian coals were calculated using the gra-

dients from the mercury intrusion curves (Figure 1) and the helium densities according to the equation

$$\text{Compressibility} = \text{unit conversion factor} \times \text{gradient} \times \text{helium density}$$

Figure 8 shows a relationship between the compressibility and coal rank of Canadian coals along with the results of Toda and Toyoda¹⁰ for comparison. Since the variation of helium density with coal rank over the range studied is comparatively small, the shape of the curve in Figure 8 is mostly determined by the change in gradient with coal rank. According to the work of Toda and Toyoda¹⁰, this change has the similar pattern with a minimum and a maximum as shown in Figure 8. In the present study, the lack of data between 80-87% carbon cannot confirm the existence of a minimum and a maximum in the relationship between the compressibility and coal rank. However, this was observed by Schuyer et al³⁸ based on sound velocity measurements, although their curve shifted slightly towards the side of higher carbon content.

SUMMARY

It has been found that the open porosity, total pore volume, micropore volume, N₂ surface area and the apparent compressibility of eleven Canadian coals decrease with decrease in the carbon content of the coal. For lignites, the majority of the pore volume is associated with the macro- and mesopores. The reactivity of coal at $\approx 1123^{\circ}\text{K}$ in an oxygen-enriched atmosphere (with or without steam present) decreases with decrease in the total pore volume and the N₂ surface area of the coal. However, this relationship does not hold at a pore volume value less than 0.070 ml g^{-1} of the coal. It is believed that both the large and small pores are important in determining the coal reactivity.

Our results agree with the phenomenon that a convex parabolic relationship exists between the helium density (minineal matter free basis) and the carbon content of the coal. This provides a simple method to estimate the helium density of a coal from its carbon and ash content.

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TABLE 1 - Chemical Analysis of Coal Samples (moisture free)

Sample (rank)	Proximate Analysis (wt %)			Ultimate Analysis (wt %)				
	Ash	Volatile Matter	Fixed Carbon ^a	C	H	S	N	O ^a
Onakawana (lignite)	26.8	40.0	33.2	49.7	3.3	5.4	0.7	14.1
Coronach (lignite)	13.4	43.5	43.1	61.1	3.6	1.1	1.0	19.8
Bienfait (lignite)	12.5	41.4	46.1	66.1	2.2	0.6	1.3	17.3
Sundance (subbituminous)	16.0	33.7	50.3	63.6	2.5	0.2	0.8	16.9
Prince (HVB)	12.5	32.9	54.6	70.7	4.9	4.9	1.4	5.8
Coalspur (subbituminous)	9.5	37.1	53.4	71.9	4.7	0.2	1.1	12.6
Balmer (MVB)	10.4	20.3	69.3	80.2	4.4	0.2	1.5	3.3
Devco (HVB)	2.8	35.4	61.8	84.7	5.6	1.3	1.3	4.3
Byron Creek (MVB)	15.3	26.2	58.5	74.4	4.3	0.8	1.2	4.0
McIntyre (LVB)	8.2	18.1	73.7	83.1	4.3	0.6	1.2	2.2
Canmore (semi-anthracite)	11.6	12.3	76.1	81.3	4.2	0.8	1.7	0.4

^a Determined by difference

TABLE 2 - Carbon Content and Some Physical Properties of coals

Coal	Cdaf (wt %)	Helium density (gm ℓ^{-1})	Mercury density (gm ℓ^{-1})	Open porosity (vol.%)	Pore volume (mlg $^{-1}$)			Pore volume (vol.%)		Gradi- ent $\times 10^5$ (mlg $^{-1}$ MPa $^{-1}$)	N ₂ Surface area (m ² g $^{-1}$)
					Total	Macro- + meso-	Micro-	Macro- + meso-	Micro-		
Onakawana	67.9	1.647	1.009	38.7	0.384	0.321	0.063	83.6	16.4	19.9	3.76
Coronach	70.6	1.504	1.281	14.8	0.116	0.069	0.047	59.7	40.3	19.9	2.09
Bienfait	75.5	1.469	1.293	12.0	0.093	0.047	0.046	50.5	49.5	16.0	1.56
Sundance	75.7	1.490	1.342	10.0	0.074	0.038	0.037	50.8	49.2	18.5	2.22
Prince	78.1	1.424	1.305	8.3	0.064	0.018	0.046	28.3	71.7	19.0	0.86
Coalspur	79.5	1.411	1.317	6.7	0.051	0.017	0.033	34.5	65.5	16.7	0.84
Devco	87.1	1.285	1.252	2.5	0.020	0.021	Nil	100	Nil	15.8	0.43
Byron Creek	87.8	1.420	1.285	9.5	0.074	0.029	0.045	39.2	60.8	14.9	0.86
Balmer	89.5	1.407	1.151	18.2	0.158	0.094	0.065	59.1	40.9	16.1	1.11
McIntyre	90.5	1.378	1.345	2.4	0.018	0.018	Nil	100	Nil	12.8	0.48
Canmore	92.0	1.440	1.358	5.7	0.042	0.029	0.013	69.8	30.2	14.1	0.76

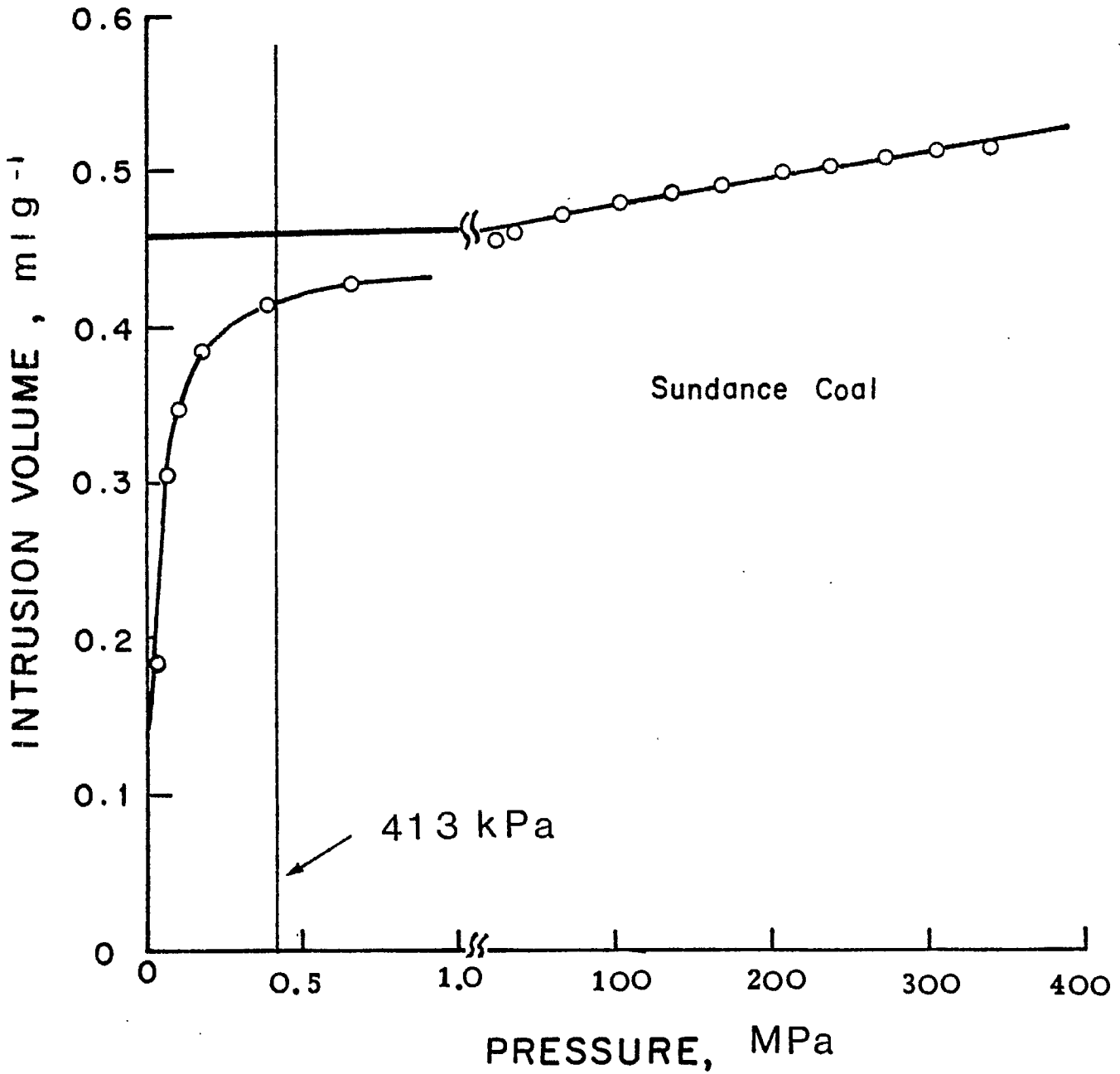


FIGURE 1 - A Mercury Intrusion Curve of Sundance Coal

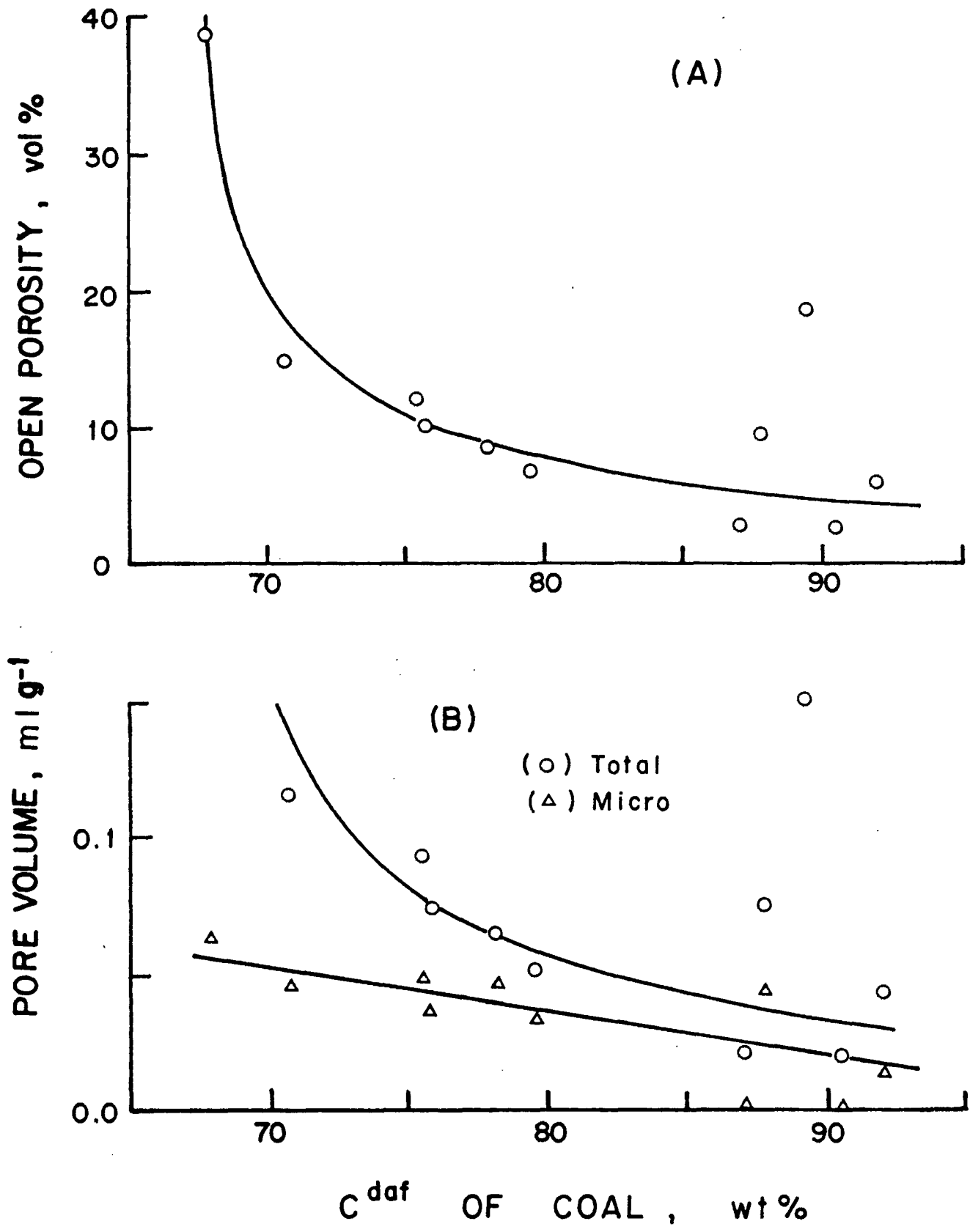


FIGURE 2 - Variation of Open Porosity (A) and Pore Volume (B) with Carbon Content of Coal

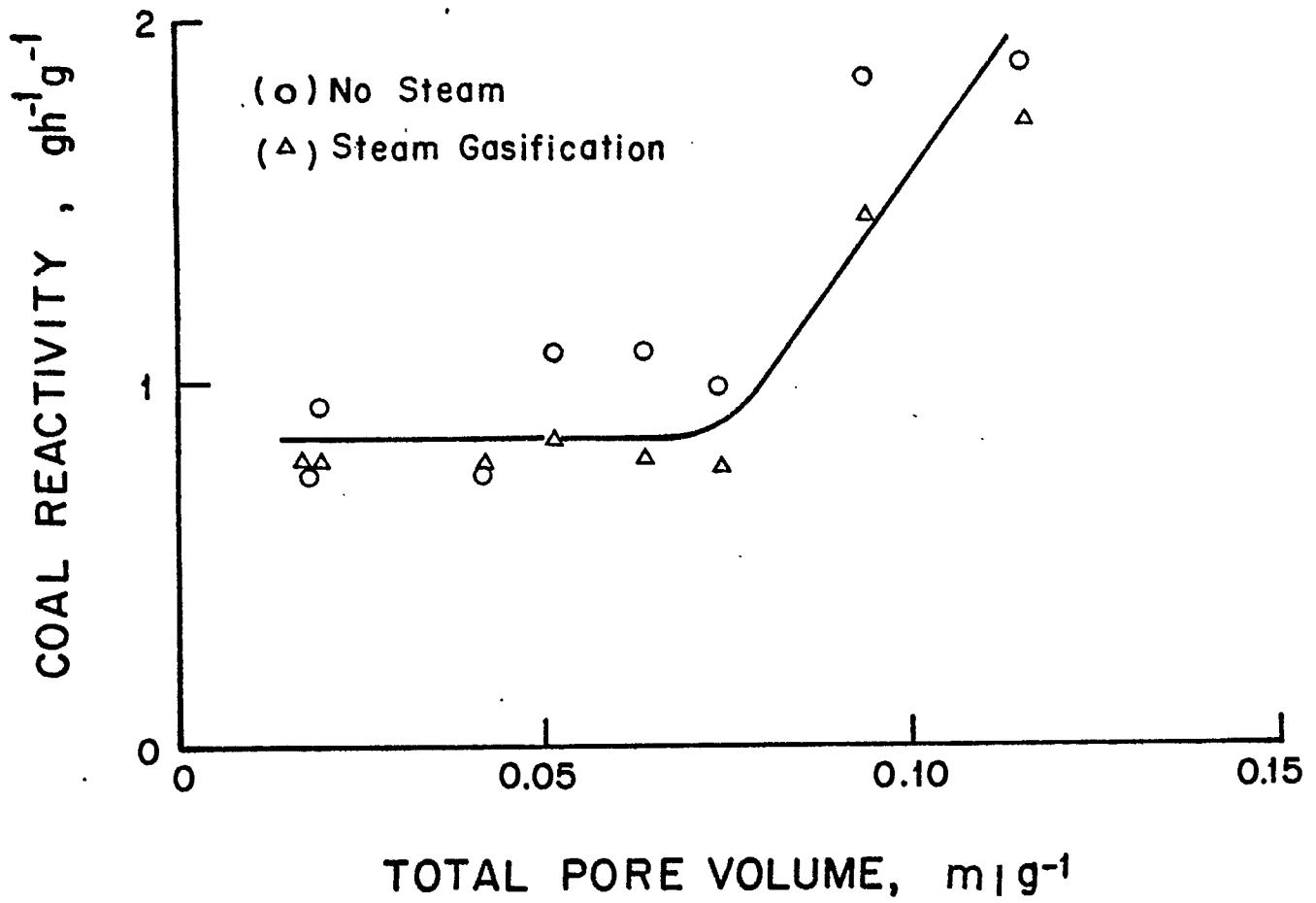


FIGURE 3 - Correlation Between Gasification Reactivity and Total Pore Volume of Coal

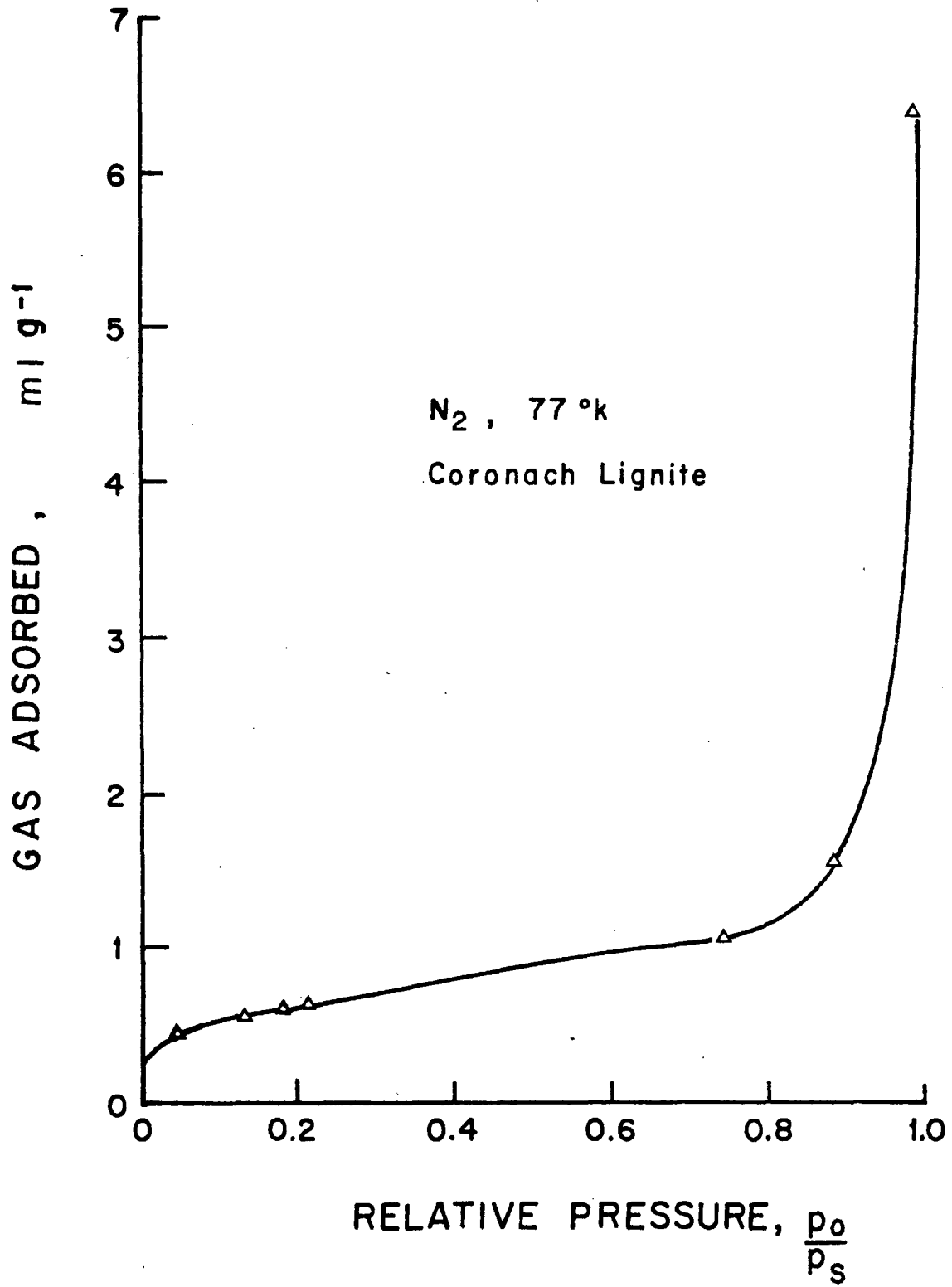


FIGURE 4 - A Gas Adsorption Isotherm of Coronach Coal

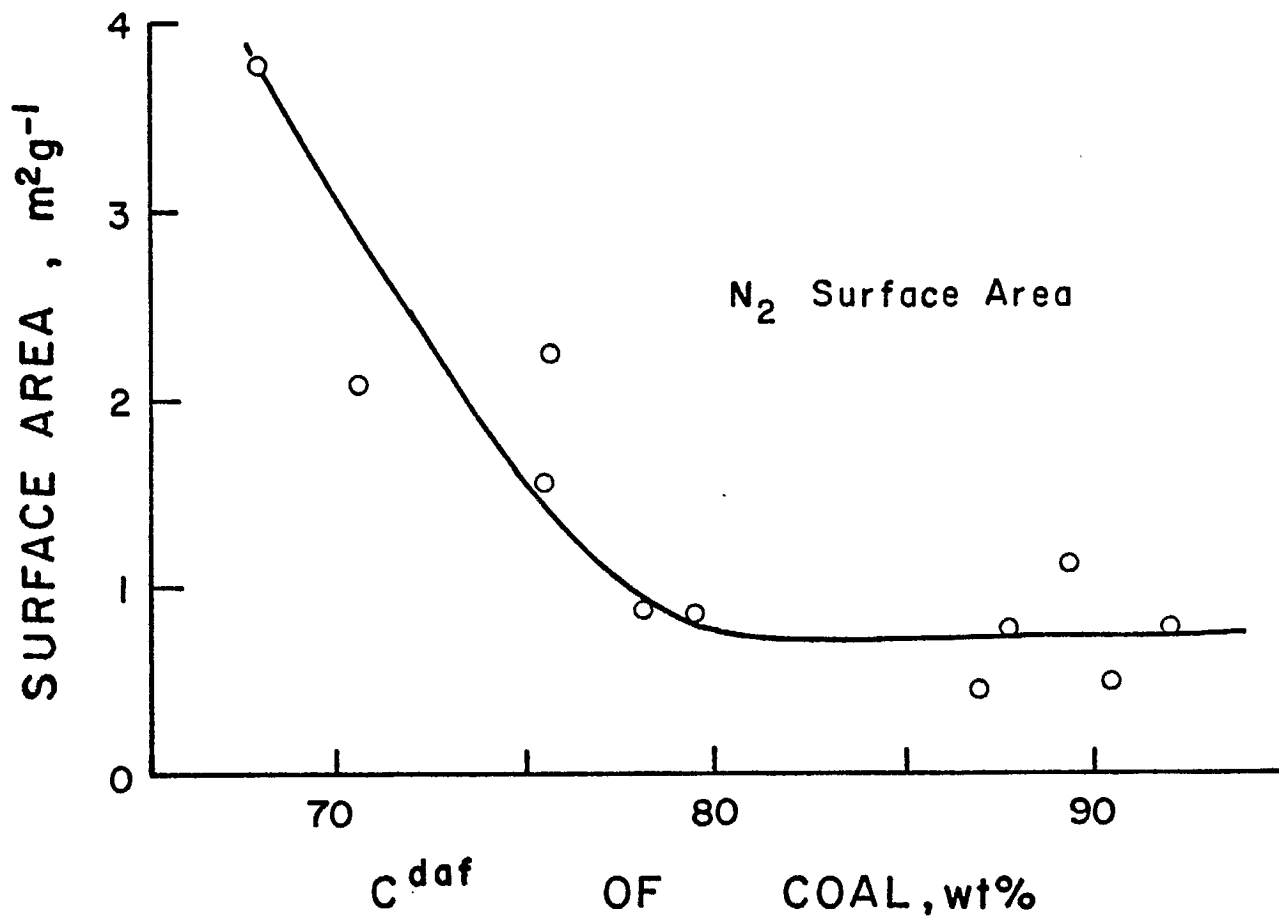


FIGURE 5 - Relationship Between Surface Area and Carbon Content of Coal

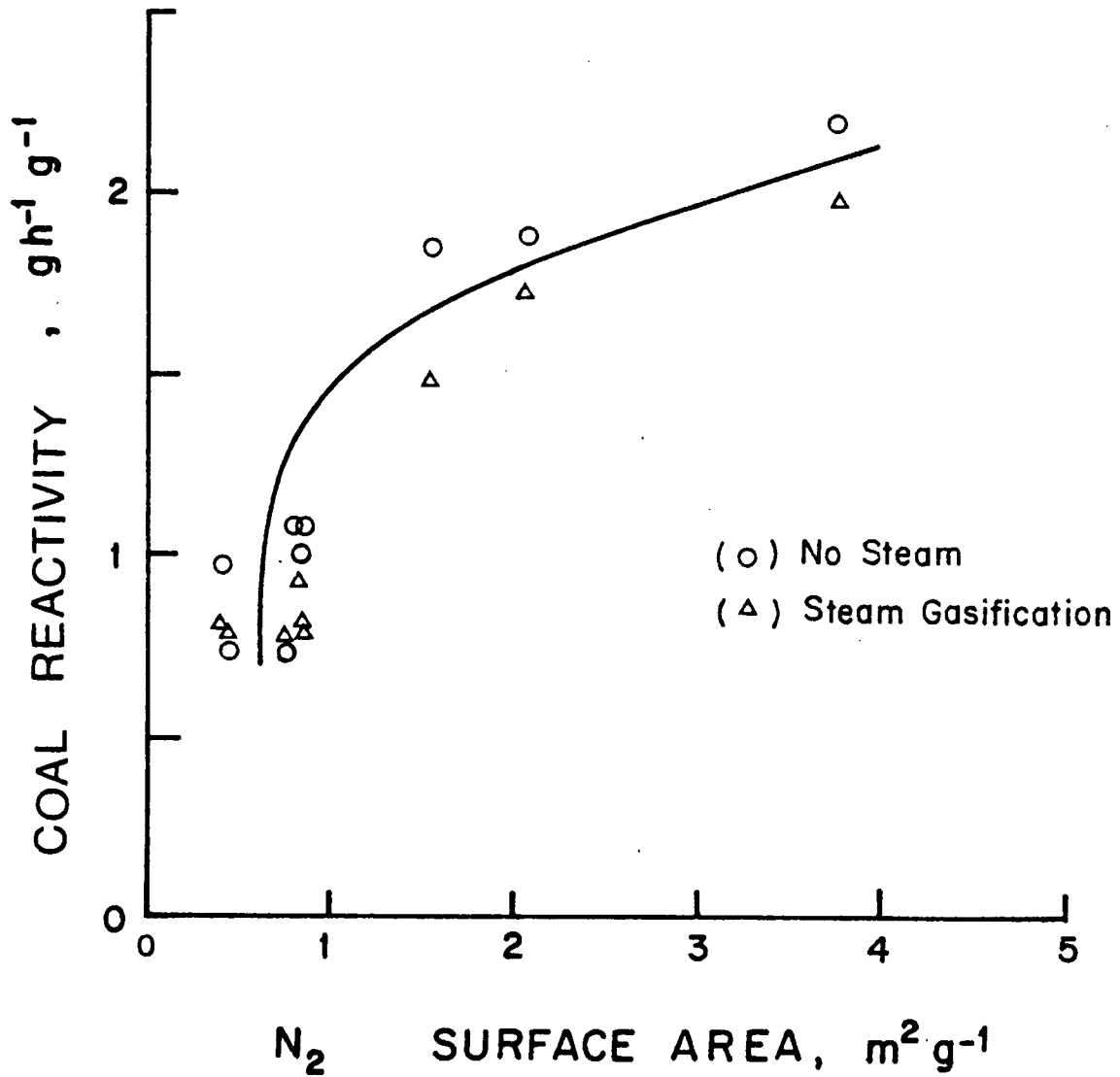


FIGURE 6 - Correlation Between Gasification Reactivity and Surface Area of Coal

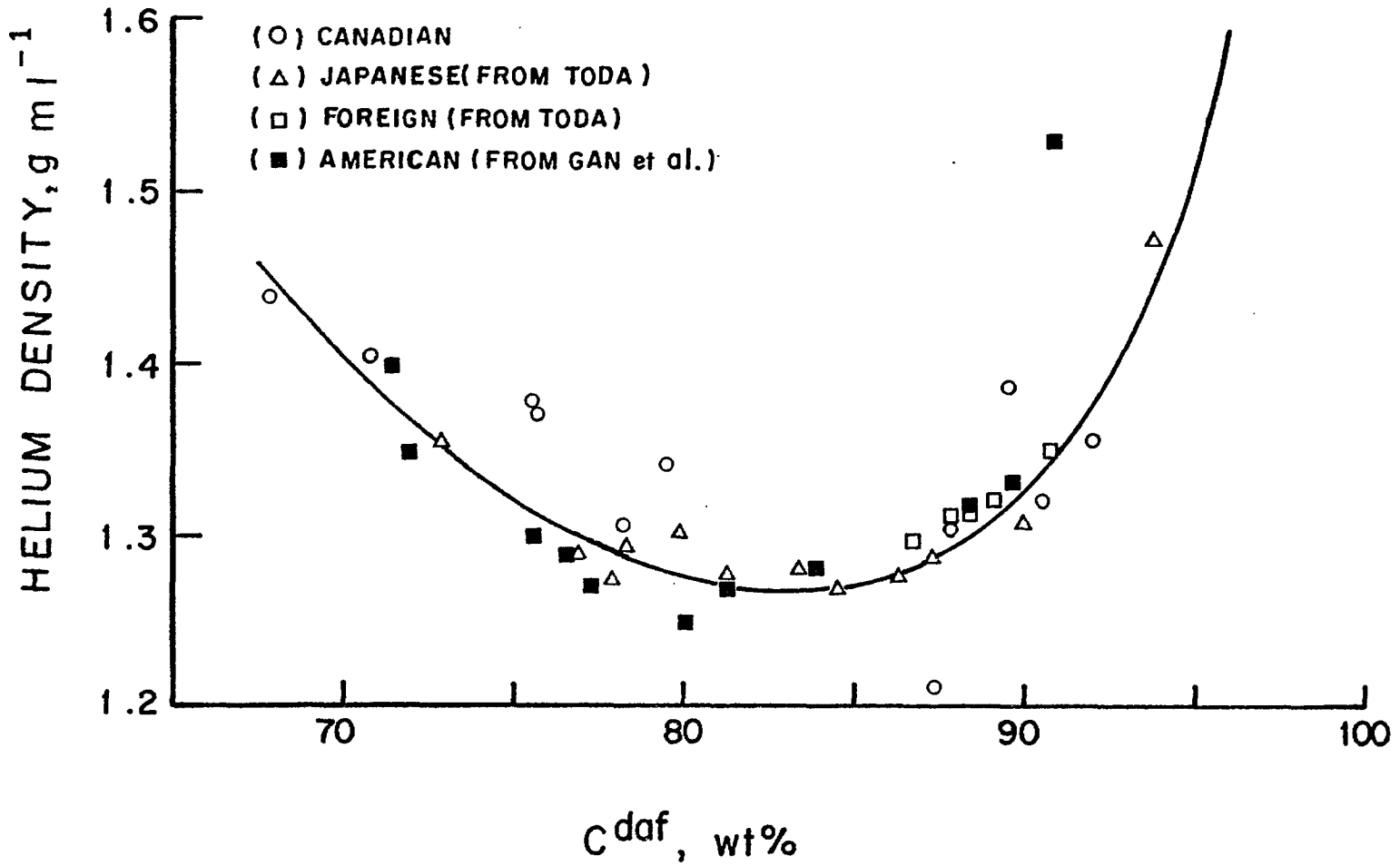


FIGURE 7 - Relationship Between Helium Density (Mineral Matter Free) and Carbon Content of Coal

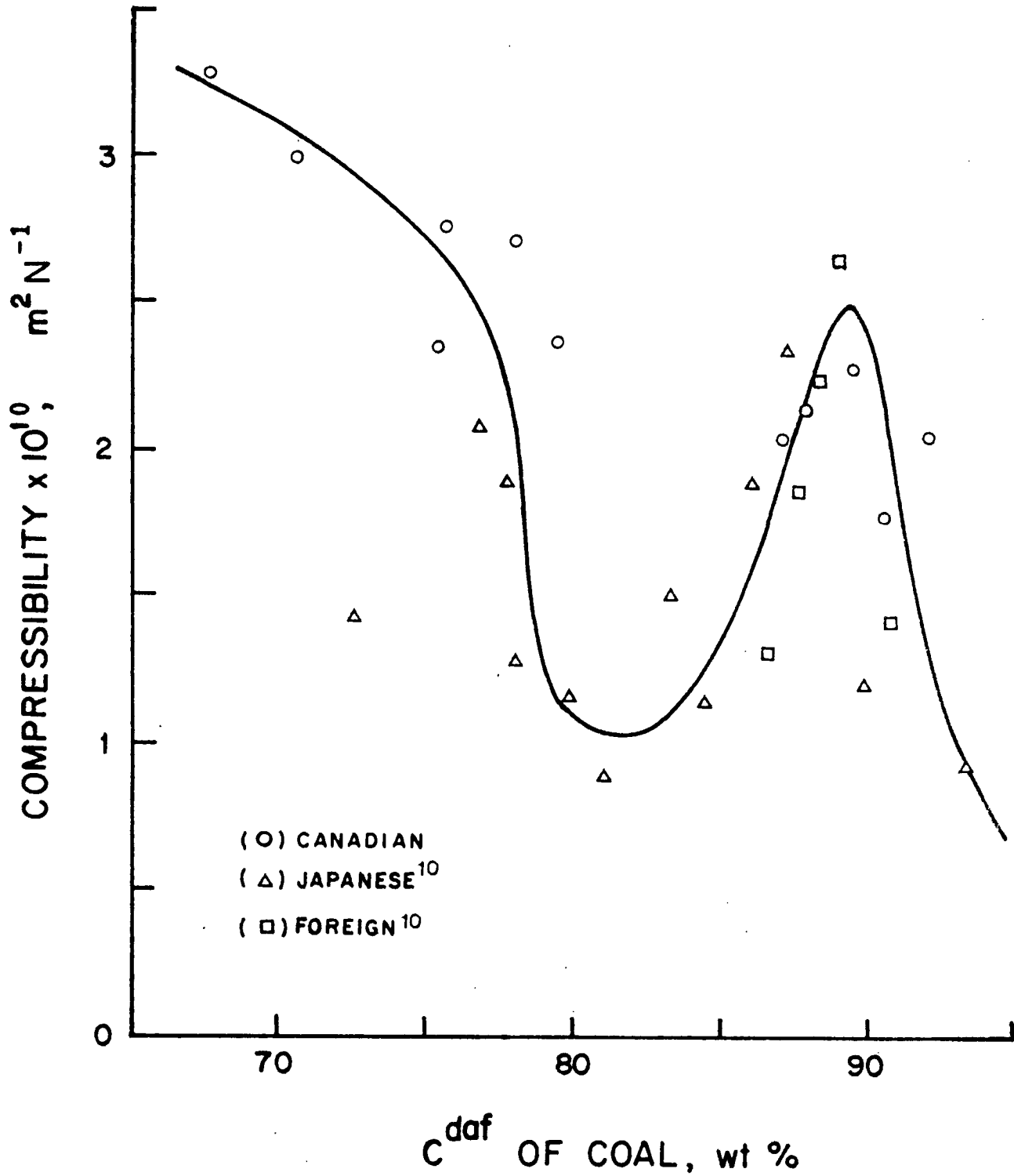


FIGURE 8 - Compressibility Versus Carbon Content of Coal