

# An Evaluation of the Significance of some Laboratory Coking Tests in Predicting the Cokeability of Coals.

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## ABSTRACT

Eleven samples of Agro-Allied, Bellview and Carbon Energy/Pittson coal blends obtained from Australia, Poland and United States of America, respectively; were subjected to basic bench scale analyses. The results obtained showed that Pittson coal (P2) has the highest Free Swelling Index of 7, while the Agro-Allied sample (A4) has the lowest FSI of 2. The highest and lowest Gieseler maximum fluidities of 4,055 ddpm and 38 ddpm were obtained for the Agro-Allied sample (A5) and Bellview sample (B2), respectively. Furthermore, Pittson sample (P2) gave the highest Ruhr total dilatation of 94%, while Agro-Allied sample (A3) yielded the lowest total dilatation of 1%.

The highest Gieseler temperature range of 109 was also determined for Pittson coal P2 and the lowest value of 60 for the Bellview coal B2. It was observed that Gieseler ratio (a parameter derived in this work from Gieseler plastometer results), Gieseler maximum fluidity, Gieseler temperature range and Ruhr total dilatation gave significant indications of sample coals' cokeability at G values above 0.97. Furthermore, the results obtained for samples with G values higher than 0.97 suggest Gieseler ratio as giving the best indication of cokeability in term of good fixed carbon and low ash. When two coals of the same source have equal FSI values, the sample with the higher temperature range will likely be more cokeable, while the sample with higher values of FSI and Gieseler temperature range were more cokeable for different FSI values.

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## INTRODUCTION

Coal can be defined as a compact stratified mass of mummified plant debris, interspersed with smaller amounts of inorganic matter and covered with sedimentary rocks. The transformation from plant tissues to coal occur due to the effects of temperature, pressure and activities of bacteria. The rank of a coal is the degree of change of the chemical composition of the coal within the series of fossil fuels from peat to anthracite (Francis and Peters, 1980). The first stage in the production of coal is thus peat, progressing through the brown coal, bituminous, and anthracite categories. The coking coal to produce metallurgical grade coke comes from the bituminous grade coals, which constitutes about 52% of the world's resources of coals. However, only about 5% of the world's supply of coals is prime coking and suitable for straight carbonization (Worldcoal, 2007; Bujnowska and Collin, 1992).

Coal is a readily combustible sedimentary rock containing more than 50% by weight or more than 70% by volume of carbonaceous materials including inherent moisture. The largest single use of coal in the steel industry is as a fuel for the blast furnace, either for the production of metallurgical coke or for injection with the hot blast. For pulverized injection, the coal must deliver a known and consistent calorific value, be reasonably low in ash and meet environmental requirement for sulphur and nitrogen oxide emissions. Only a certain class of coals possessing very specific properties and composition are suitable for the production of a metallurgical grade coke (Worldsteel, 2007).

The prediction of coke strength from the results of laboratory tests on coal is of great importance in coke-making. The free crucible swelling test is widely used as a general indication of the

suitability of a particular coal for coke-making in by-product ovens. For example, coals with a Crucible Swelling Number (CSN) of less than about 4 are unsuitable for metallurgical coke-making and those with CSN of 4½ to 9 will produce metallurgical grade coke of various degrees of hardness. However, no direct correlation could be found between CSN and coke strength because coals of the same swelling number but of different volatile matter produces cokes of widely different strengths.

The dependence of the swelling of coal on the heating rate and the need to measure the property under conditions approximating to those of industrial practice led to the development of an important group of tests in which the coal was heated for all or part of the test at heating rate of 1 to 3°C/minute (Willkinson, 1971). Contraction and dilatation tests, which include Ruhr dilatometry and Gieseler plastometry are other important plastic tests. The tests determine the temperatures at which coals exhibit contraction and swelling behavior. Coal quality is now being predicted with considerable accuracy from the dilatation characteristics of the coal blends. Simonis developed a mathematical expression to determine the coking capacity, G for coal blends (Afonja, 1991; Beck, 1984):

$$G = \frac{E + V}{2} \times \frac{c + d}{V \times c + E \times d} \quad (1)$$

where,

G = Coking capacity

E = Softening temperature (K)

V = Temperature of maximum dilatation

c = % Maximum contraction

d = % Maximum dilatation

The aim of this research work is to establish the significance of free swelling index, Gieseler plastometry and Ruhr dilatometer bench scale coking tests in predicting the cokeability of coals for metallurgical coke-making.

## MATERIALS

Drums of the Australian Agro-Allied, Polish Belview, American Carbon Energy coals, and several kilograms of the American Pittson Coal supplied to the National Metallurgical Development Centre (NMDC), Jos, Nigeria, were used as experimental samples.

## METHODS

### Sample Collection

The samples were collected from the coal fields of Australia, Poland and United States and supplied to Nigeria.

### Sample Preparation

The Agro-Allied, Bellview, and Carbon Energy coals were supplied in drums with varying size consist. Each coal supply was made into a heap that was after spread over the pilot plant floor. Samples were then collected randomly at various points of the spread coal. The collected sample was then thoroughly mixed, air dried over night and crushed for the various analyses. The few kilograms of Pittson sample was also air dried overnight and then crushed to the desired sizes.

### Proximate Analysis

The proximate analysis was carried out with modified forms of methods described in Francis and Peters (1980). It involved the determination of moisture, volatile matter, ash, and fixed carbon contents of the coal.

### Moisture Content

The crucible was preheated in an oven at a temperature of 110°C for 1 hour. One gram of coal ground to pass 250 µm was then heated in the crucible at 110°C for 1 hour. The percent loss in weight accounted for the moisture content and it was calculated with the formula:

$$\% \text{ Moisture} = \frac{W_i - W_f}{W_i} \times 100 \quad (2)$$

$W_i$  = initial weight of sample

$W_f$  = final weight of sample after the moisture test

### Volatile Matter

The silica crucible was pre-heated in the Carbolite 11/81/1083 muffle furnace for 7 minutes at 900°C and cooled. About 1 g of the coal sample ground to pass 250  $\mu\text{m}$  was then placed in the crucible and heated in the muffle furnace for about 7 minutes at 900°C. The weight of the heated sample was then determined. The percent loss in weight gave the volatile matter content of the coal and it was calculated with the formula:

$$\% \text{ Volatile matter} = \frac{W_i - W_f}{W_i} \times 100 \quad (3)$$

where:

$W_i$  = initial weight of sample

$W_f$  = final weight of sample after the volatile matter test

### Ash Content

The silica crucible was pre-heated in muffle furnace at 825°C for 1 hour. It was then cooled and weighed. 1 g of the coal sample ground to pass 250  $\mu\text{m}$  was then placed in the crucible (with the lid on) and heated in the furnace at the same pre-heating temperature for 1 hour. The crucible was then cooled in the desiccators and weighed. The incombustible residue of the coal was then weighed. The percent ash content was calculated from the weight of the incombustible residue with the formula:

$$\% \text{ Ash} = \frac{W_r}{W_i} \times 100 \quad (4)$$

$W_i$  = initial weight of sample

$W_r$  = weight of ash residue after the ashing test

### Fixed Carbon

Fixed carbon of the coal was determined by calculation with the relation:

$$\% \text{ Fixed carbon} = 100 - \% \text{ moisture} - \% \text{ Ash} - \% \text{ Volatiles} \quad (5)$$

### Free Swelling Index

The Free Swelling Index test was conducted with modified form of method described in ASTM D720-67. One gram of the coal sample, ground to pass 250  $\mu\text{m}$  was weighed into a silica crucible. The crucible and its content were placed on wire gauze set on a tripod stand. The crucible was then heated on Bunsen burner flame for 7 minutes. The coke button obtained was thereafter compared with standard profiles and the appropriate free swelling numbers determined for each coal sample.

### Gieseler Plastometry

The Gieseler plastometry test was carried out in substantial in compliance with ASTM standard D2639-74. The Fuel Research Company 420001A4 Gieseler plastometer was used for the test. About 5 g of the coal sample ground to pass 425  $\mu\text{m}$  was compressed by a 10 kg weight placed on the sample with the stirrer arms fully embedded. After assembling the apparatus, the sample was heated as programmed on the Gieseler programmer. The stirrer was immobile at the start of the heating since the rabble arms were rigidly held by the compressing coal. The movement started when the coal softened and continued as long as the coal remained in the plastic state. The movement stopped when the coal re-solidified. The speed of rotation (in dial divisions per minute (ddpm)) varies with the fluidity of coals. The parameter, Gieseler ratio, was derived from the Gieseler properties with the formula:

$$GR = \frac{GTR}{\text{Log}(GMF)} \quad (6)$$

Where:

GR = Gieseler ratio

GTR = Gieseler temperature range

GMF = Gieseler maximum fluidity

capacity for each coal was then calculated with Equation 1.

### Ruhr Dilatometry

The dilatometric characterization of the coals was carried out with the German DIG 2\_05 Ruhr dilatometer. In the test, the variation in the length of a column of coal during heating was measured. The coal sample (ground to pass 250 µm) was compacted into a pencil form. The pencil of coal was then placed in a metal tube and a piston rod was inserted into the tube to rest on piston's top. The other end of the piston rod was attached to a rotating barrel to record the vertical movement of the piston. On heating, the column of coal softened and contracted in length due to the plastic deformation under the action of piston.

When the coal pencil softened, bubbles of gas were formed due to decomposition of the softened coal mass. The gases released were trapped causing the coal column to swell up. The softening temperature, temperature of maximum dilatation, the percentages maximum contraction and dilatation were determined. The G-coking

### RESULTS

The results of all the analyses are presented in Table 1.

### DISCUSSION

For the various coals tested, it was observed that the values of Free Swelling Index (FSI) do not have a clear effect on the coke strength in terms of Simonis' G-value. For the Agro-Allied samples, the highest G-value was obtained for sample A1 with an FSI value of 2.5, while the Carbon Energy sample C1 with a higher FSI of 4 gave a lower G-value of 1.01. However, for the Pittson coal, sample P2 with the higher G-value of 7 gave the higher G-value of 1.04. The latter result agree with the results obtained for some German coals with FSI values of 2.5, 3 and 4 which gave increasing G-values of 0.261, 0.714 and 0.929, respectively (Weskamp *et al.*, 1992).

**Table 1:** Proximate, Gieseler Plastometer, and Ruhr Dilatometer Analyses Results for Agro-Allied, Bellview, Carbon Energy, and Pittson Coals.

Parameters	A1	A2	A3	A4	A5	B1	B2	C1	C2	P1	P2
Moisture%	1.09	1.62	1.90	1.20	0.90	1.50	0.80	1.50	1.30	0.30	0.50
Volatile matter (daf)	29.87	29.60	31.40	29.70	30.20	31.80	31.30	22.60	33.70	26.60	27.20
Ash(db)	8.62	9.40	8.40	9.50	9.70	5.80	6.10	2.70	5.20	5.70	6.40
Fixed carbon(daf)	70.10	70.40	68.60	70.40	69.80	68.20	68.70	77.30	66.40	73.40	72.90
Free swelling index(FSI)	2.5	3.5	2.5	2	2.5	2.5	2.5	4	2.5	5.5	7
Maximum dilatation (%) (RD)	-5	-5	-27	-7	-8	10	-7	40	56	41	72
Maximum contraction (%) (RMC)	30	26	28	27	22	26	24	28	30	23	22
G-Value	0.94	0.92	0.36	0.91	0.93	0.97	0.93	1.01	1.02	1.02	1.04
Total dilatation (%) (RTD)	25	21	01	20	14	36	17	68	86	64	94
Gieseler maximum fluidity(GMF)	243	501	3860	720	4055	48	38	50	2346	433	2218
Gieseler Temp Range (GTR)	86	84	89	84	98	70	60	91	94	82	109
Gieseler Ratio (GR)	36.1	31.1	24.81	29.4	27.2	41.6	38	53.6	27.9	31.1	32.6

Legend:

A = Agro-Allied coal (samples A1, A2, A3, A4 and A5)

B = Bellview coal (samples B1 and B2)

C = Carbon Energy coal (samples C1 and C2), P = Pittson coal (samples P1 and P2)

For the two Bellview samples with equal FSI values, different G-values of 0.93 and 0.97 were obtained. It was observed for all the samples that lower G-values were only obtained at increasing FSI values when the Gieseler temperature range and total dilatation percent are lower than for coal samples with lower FSI values. These results suggest that G-value increases with increasing FSI, particularly when the samples being considered have comparatively high Gieseler temperature range and total dilatation percent.

For the Agro-Allied samples, the G-values increase generally with increasing Ruhr total dilatation percent, except for samples A4 and A5. The lowest G-value occurs at the least total dilatation of 1%. For the Bellview samples, sample B1 with the higher total dilatation percent of 36% gave the highest G-value of 0.97 as against 0.93 obtained for sample B2 with total dilatation of 17%. For the Carbon Energy samples, the higher G-value of 1.02 occurs at total dilatation of 86% as against 1.01 obtained at 68% total dilatation. For the Pittson samples, sample P2 with the higher total dilatation of 109 gave the higher G-value of 1.04 as against 1.02 for sample P1 with total dilatation of 82%. These results strongly suggest that G-values of coals generally increase with increasing total dilatation irrespective of the coal source.

It was also observed that the G-value has no clear dependence on Gieseler temperature range and Gieseler maximum fluidity for all the samples tested except the Carbon Energy sample C2 and Pittson sample P2 with high G-values of 1.01 and 1.04 corresponding to high Gieseler temperature range and Gieseler maximum fluidity 94, 2346 ddpm and 109, 2218 ddpm; respectively. The very high Gieseler temperature range and Gieseler maximum fluidity of 89 and 3860 ddpm for sample A3 however gave a much lower G coking value of 0.36.

For the Agro-allied samples, sample A5 gave the highest Gieseler maximum fluidity of 4,055 ddpm, while sample A3 gave 3,860 ddpm. It was observed that the Gieseler maximum fluidity at the FSI value of 2.5 for A5 exceeds the Gieseler maximum fluidity of 501 ddpm for sample A2 with FSI value of 3.5. The Bellview samples B1 and B2 with FSI value of 2.5 gave Gieseler maximum fluidity of 48 ddpm and 38 ddpm, respectively. The Carbon Energy sample C1 with the higher FSI of 4 gave a maximum fluidity of 50 ddpm as compared to 2,346 ddpm by sample C2 with an

FSI of 2.5. The Pittson sample with the higher FSI of 7 gave a maximum fluidity of 2,218 ddpm, while sample P2 with FSI of 5.5 gave a lower Gieseler maximum fluidity of 433 ddpm.

These results agree with the results obtained by Parthasarathy *et al.* (1992) for Indian Gidi and Rajrappa samples. The two coals gave FSI of 1.5, while the medium coking Rajrappa coal gave a maximum fluidity of 114 ddpm and the Gidi sample gave a much higher maximum fluidity of 217 ddpm. Also, the prime grade Kunidih sample gave an FSI of 5 and a fluidity which far exceeds the maximum fluidity of 662 ddpm for the Rhojudih coal sample with an FSI of 3.5 which is only slightly lower than for the former. The two prime grade Indian Patherdih and Sudamih samples that both have FSI of 2.5 gave maximum fluidity of 516 ddpm and 582 ddpm, respectively. For coals carbonized in the defunct Czechoslovakia, an imported sample with an FSI of 8 gave a Gieseler maximum fluidity of 268 ddpm, while medium coking captive coal with an FSI of 3 gave a much higher maximum fluidity of 2,455 ddpm (Buchtele *et al.*, 1992).

Also, the low volatile American coal carbonized in France having an FSI of 8 to 9 gave maximum fluidity that vary between 50 and 150 ddpm, while medium volatile Polish samples with FSI of 7 to 8 gave Gieseler maximum fluidity of 500 to 1,500 ddpm (Garin *et al.*, 1987). These results show that while coals with high FSI may generally have good Gieseler maximum fluidity, there is no clear relationship between FSI values and Gieseler maximum fluidity of coals. Also, the G-values of coals do not correspondingly improve with the Gieseler maximum fluidity values.

For the Agro-Allied samples, the highest temperature ranges of 89 and 78 were obtained for samples A3 and A5 with FSI of 2.5, while sample A2 with the highest FSI of 3.5 gave the same temperature range of 84 with sample A4 with a lower FSI of 2.5. For Bellview samples, temperature ranges of 70 and 60 were obtained for samples B1 and B2 (with the same FSI of 2.5), respectively. For the Carbon Energy samples, a higher temperature range of 86 was obtained for sample C2 with the lower FSI of 2.5 as compared to sample C1 with the higher FSI of 4. As regards Pittson samples, sample P2 with the higher FSI of 7 gave the higher temperature range of 94 as compared to 64 for sample P1 with an FSI of 5.5. For a Polish ortho-coking coal having an FSI of 5.5, the temperature range of 72 was obtained.

When 5% coal tar pitch was added to the latter, the FSI increased to 6 and temperature range was raised to 98 (Bujnowska and Collin 1992).

It was also observed that when two coals of the same source have the same FSI, the one with the higher temperature range gives the higher G-value. Furthermore, when the FSI of two coals of the same source differ, the one with the higher temperature range gives the higher G-value when its FSI is higher. These results show that coke strength generally increases with Gieseler temperature range values and that the FSI values of coals have no clear relationship with their temperature ranges. However, Gieseler temperature range parameter provides an additional guide in grading coals of the same source but different FSI values.

For the Agro-Allied samples, sample A1 with the highest Gieseler ratio of 36.1 gave the highest G-value of 0.94, while sample A4 with the least Gieseler ratio of 24.8 gave the least G-value of 0.36. For the Bellview samples, sample B1 with the highest Gieseler ratio of 41.6 gave the higher G-value of 0.93. For Pittson coal samples, sample P2 with the higher Gieseler ratio of 32.6 gave the higher G-value of 1.04 as compared to 1.02 given by sample P1 with the Gieseler ratio 31.1. For Carbon Energy samples, both samples gave almost the same G-value despite the difference in their Gieseler ratios. The slight deviation from the observed trend in samples A2 and A5 may be due to the much higher Gieseler maximum fluidity of A5. For coal samples carbonized in Germany, samples with FSI of 2.5, 3, and 4 gave G-values of 0.261, 0.714, and 0.929, respectively (Weskamp *et al.*, 1992). G-values have been shown to give a good prediction of coke micum strength (Wilkinson, 1971). These results strongly suggest that the derived Gieseler ratios give a fairly accurate prediction of coke strength in terms of G-values for coals notwithstanding their sources. The least G-value occur for sample A3 despite its very high Gieseler maximum fluidity of 4,055 ddpm and high temperature range of 89. It is observed that the least Ruhr maximum dilatation and total maximum dilatation occurred for sample A3. This result thus indicates that a very low Ruhr maximum and total dilatation percent may lead to a poor grade coke.

The highest Simonis' G-coking capacity of 1.04 was obtained for Pittson sample P2 with the highest total dilatation percent, Gieseler temperature range, Ruhr dilatation percent and

Free Swelling Index of 94, 109°C, 72 and 7; respectively. The sample also gave the one of the two lowest Ruhr maximum contraction percent of 22, the second sample with the same maximum being Agro-Allied sample A5. Furthermore, the Pittson sample P2 gave the second lowest moisture content of 0.5%, third lowest volatile matter (daf) of 27.20%, the fourth lowest Gieseler ratio of 32.6, sixth lowest ash content of 6.40%, third highest fixed carbon (daf) of 72.90% and the third highest Gieseler maximum fluidity of 2,218 ddpm.

The second highest Simonis' G-coking capacity of 1.02 was obtained for Carbon Energy C2 and Pittson sample P1. The C2 sample gave the highest Ruhr contraction percent (the same with Agro-Allied A1 with G value of 0.94), second highest total dilatation percent, third highest Gieseler maximum fluidity, third highest Gieseler temperature range and second highest Ruhr dilatation percent of 30, 86, 2346 ddpm, 94°C and 56; respectively. The sample P1 also gave the lowest moisture content, second lowest volatile matter (daf), third lowest ash(db), second highest fixed carbon (db), second highest Free Swelling Index, second lowest maximum contraction and fourth lowest Gieseler ratio of 0.30%, 26.60%, 5.70%, 73.40%, 5.5, 23% 31.1; respectively.

The fourth highest Simonis' G-coking capacity of 1.01 was obtained for Carbon Energy sample C1. The sample gave lowest ash percent of 2.70, and the highest fixed carbon percent and Gieseler ratio of 77.30 and 53.6, respectively. Also, the sample gave the third highest FSI, Ruhr dilatation, Ruhr contraction and Ruhr total dilatation of 4, 40%, 28%, 68%, and third lowest Gieseler maximum fluidity of 50 ddpm. In addition, the sample yielded the fourth highest Gieseler temperature range of 94. For the samples with G values in the range 1.01 to 1.04, the G value generally increases with increasing gieseler temperature range and Ruhr total dilatation. Samples with the high Gieseler fluidities of 2346 ddpm and 2218 ddpm were found to produce the highest G values of 1.02 and 1.04, respectively. The results obtained also showed that G coking value generally increase with decreasing ash content and the G value of 1.01 corresponding to the highest fixed carbon occurred for the sample with the least ash content of 2.70%. It was further observed that G coking value has no clear dependence on volatile matter content.

For the samples A1, A2, A3, A4, A5, B1 and B2 with G values lower than 1.01, it was observed that G value generally increase with increasing total Ruhr dilatation percent except for the deviation obtained in the total Ruhr dilatation percent for the same G value of 0.93 for both Agro-Allied and Bellview samples A5 and B2, respectively. The lower Ruhr total dilatation percent of 14 obtained for A5 may be due to its higher ash content of 9.70% as against 6.10% for B2. The results also showed that the G value is also directly proportional to Gieseler ratio, except for Agro-Allied A1 and A5 samples.

The deviation noted may be due to the high ash contents percent of 8.62 and 9.70 for A1 and A5, respectively. It was further observed that samples A5 and A3 with very high Gieseler maximum fluidity of 4055 ddpm and 3860 ddpm produced low G values of 0.93 and 0.36, respectively; while sample B1 with the lowest Gieseler maximum fluidity of 48 ddpm and highest Gieseler ratio of 41.6 gave the highest G value of 0.97 for the range 0.36 to 0.97. The results obtained for samples C1, C2, P1 and P2 with G values higher than 0.97 also showed that sample C1 with the highest Gieseler ratio of 53.6 and lowest Gieseler maximum fluidity has the highest fixed carbon and lowest ash content.

In summary, it was observed that Gieseler ratio, Gieseler maximum fluidity, Gieseler temperature range and Ruhr total dilatation gave significant indications of sample coals' cokeability at G value above 0.97. However, the results obtained for the samples with G value higher than 0.97 suggest Gieseler ratio as giving the best indication of cokeability in term of good fixed carbon and low ash. For the samples with G values lower than 1.01, it was also observed that a combination of Gieseler ratio and Ruhr total dilatation parameters produced the best indication of cokeability.

## CONCLUSIONS

The eleven coal samples from Australia, United States of America and Poland were subjected to the bench scale quality analysis. The results obtained showed that the derived Gieseler ratio gives a fairly accurate prediction of coke strength at G values above 0.97 in terms of high fixed carbon and low ash; while a combination of Gieseler ratio and Ruhr total dilatation parameters produced the best indication of cokeability at G values lower than 1.01. It was observed that G-

values and hence coke strength generally increases with Ruhr total dilatation percent, Gieseler temperature range and FSI values, particularly when the sample has comparably higher temperature range and Ruhr total dilatation percent. It has been observed that there is no clear relationship between the FSI values of coals and their temperature ranges and Gieseler maximum fluidity. When two coals of the same source have equal FSI values, the sample with the higher temperature range will likely be more cokeable, while the sample with higher values of FSI and Gieseler temperature range will be more cokeable for different FSI values.

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