CANADA

AN INDEX OF ASH CLINKERING AND THE INFLUENCE OF ADDITIVES ON EASTERN CANADIAN COALS

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AN INDEX OF ASH CLINKERING AND THE INFLUENCE OF ADDITIVES ON EASTERN CANADIAN COALS

by

G. K. Lee* and J. Z. Skulski**

SUMMARY

This Technical Bulletin describes experiments carried out to determine the effect of additives on the clinkering and caking properties of a Cape Breton coal named "Dominion". The results of numerous crucible-scale tests and combustion-rig tests with various additives are then compared with values obtained with untreated coal.

It was established that the ASTM ash-softening temperature is not a good measure of the clinkering characteristics of Dominion coal when treated with additives. It was found, instead, that acceptable criteria are the ASTM ash-fusibility range, the clinker density, and the clinker impact strength. A consistent relationship between fusion temperature and ash composition was obtained by grouping oxide components of the ash in a particular ratio which has been named the "ash index".

To better understand the mechanism of clinker formation, the mineral compositions of three selected clinker samples were determined by petrographic examination and X-ray diffraction analysis.

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Direction des Mines

Bulletin Technique TB 19

UN INDICE DE FORMATION DU MÂCHEFER - EFFET D'ADDITIFS SUR LES HOUILLES DE L'EST DU CANADA

par

G.K. Lee* et J.Z. Skulski**

RÉSUMÉ

Ce Bulletin Technique décrit les résultats d'essais, entrepris pour déterminer l'effet d'additifs sur les propriétés de formation de mâchefer et d'agglutination d'une houille du Cap-Breton appelée "Dominion".

De nombreux échantillons, avec et sans additifs, ont été soumis à des essais à l'échelle du creuset et à l'appareil de combustion. On a comparé les caractéristiques de mâchefer et d'agglutination ainsi obtenues avec celles du charbon pur.

On a établi que la température de ramollissement des cendres (ASTM) n'est pas une bonne mesure des caractéristiques de formation de mâchefer du charbon Dominion traité avec des additifs. A sa place, on a trouvé que des critères acceptables sont le niveau de fusibilité des cendres (ASTM), la densité du mâchefer et la résistance aux chocs du mâchefer. Une relation ferme entre la température de fusion et la composition des cendres a été obtenue en groupant les composants oxygénés des cendres dans un certain rapport, qui a reçu le nom d' "indice de cendres".

Pour mieux comprendre le mécanisme de formation du mâchefer, la composition minéralogique de trois échantillons de mâchefer choisis a été determinée par examen pétrographique et analyse de diffraction aux rayons X.

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INTRODUCTION

A cooperative research program between the Fuels and Mining Practice Division, Mines Branch, Department of Mines and Technical Surveys, and the Dominion Steel and Coal Corporation Limited, was initiated in January 1959 to develop methods of improving the combustion performance on conventional stoker grates of a Cape Breton coal named "Dominion". The part of the research program with which this technical bulletin deals is an investigation of the benefits of additives on the burning properties of Dominion coal through reducing its caking tendency and raising its ash-fusion temperature. In this investigation experiments have been limited to 1-1/4 in. x 0 nut-slack size of Dominion coal. The results are described herein. The apparatus and procedures employed were described at the Eleventh Dominion-Provincial Coal Research Conference.(1) 1/

ASH-FUSION DETERMINATION

Throughout the technical bulletin reference is made to ash-fusion properties and determinations. The determination of fusibility of coal ash consists of melting cones of the ash to a fluid state under specified conditions. The standard conditions specified by the American Society for Testing Materials (ASTM) include a reducing atmosphere in the furnace and a definite temperature cycle.

1/ References are listed at the end of the bulletin in the order in which they are numbered in the text.

During the test three temperatures are recorded:

1. Initial deformation temperature. This is the temperature at which the points of the cones first become round.
2. Softening temperature. This is the temperature at which the cones melt to a spherical lump.
3. Fluid temperature. This is the temperature at which the cones become flat molten masses.

The ash-softening temperature is universally employed for comparison purposes and it is often incorrectly called the ash-fusion temperature. To prevent any confusion a clear distinction is made between the two terms in this technical bulletin.

RELATIONSHIP OF ASH-SOFTENING TEMPERATURE TO ASH COMPOSITION AND FURNACE CONDITIONS

Correlation by Ash Index

When the coal-additive investigation commenced it was known that the caking tendency of Dominion coal could be reduced by an inert material if applied in sufficient quantity; therefore, most of the laboratory studies were oriented to control of ash-fusion properties, especially softening and fluid temperatures.

Preliminary studies clearly established that the results of research on United States coals, relating the thermal and chemical properties of the ash, could not be applied to Dominion coal. Thus, it was necessary to develop a distinctive means of correlating softening temperature with composition of ash from various mixtures of additive and Dominion coal. A consistent relationship was obtained only after the oxide components of the ash were grouped into a ratio which has been named the "ash index". This index is calculated by dividing the sum of the silica and alumina components of the ash by the sum of the principal oxides in the ash.

An ash analysis of Dominion 1-1/4 in. x 0 nut-slack coal is given in Table 1; the silica and alumina total 58.5% and the sum of all the oxides in this case is 98.9%. Therefore, the ash index for this particular ash analysis is 0.591.

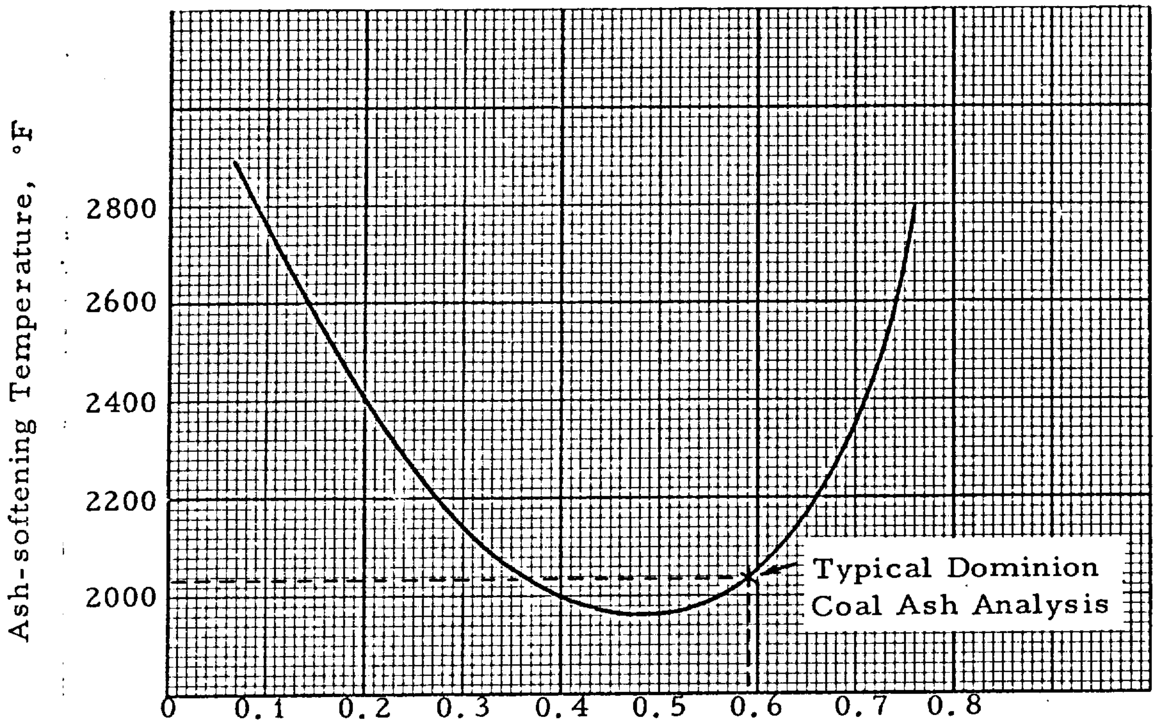
TABLE 1

A Typical Ash Analysis of Dominion 1-1/4 in. x 0 Nut-Slack Coal

SiO_2	%	32.4
Al_2O_3	%	26.1
Fe_2O_3	%	33.5
CaO	%	4.5
SO_3	%	1.17
Na_2O	%	0.05
MgO	%	0.28
K_2O	%	0.94

Having established a correlation, selected materials were added to the coal in sufficient amounts to yield a range of ash indices from 0.1 to 0.8, and for each sample the ASTM ash-softening temperature was plotted against ash index. The resulting average curve is shown in Figure 1. The "X" on the curve marks the position of the typical untreated coal ash described in Table 1.

From Figure 1 it can be seen that the ash index of Dominion coal may be decreased by the addition of lime or iron oxide. Unfortunately, small quantities of these oxides decrease the ash-softening temperature, but as larger quantities are added the ash-softening temperature increases although the ash index continues to decrease. On the other hand, both the ash index and ash-softening temperature may be increased by the addition of silica and alumina in any quantity.



$$\text{Ash Index} = \frac{Al_2O_3 + SiO_2}{CaO + Fe_2O_3 + SO_3 + Al_2O_3 + SiO_2 + Na_2O + K_2O + MgO}$$

Figure 1. Relation between ash index and ash-softening temperature.

Effect of Oxidizing Atmosphere on Softening and Fluid Temperatures of Ash

A series of laboratory ash-fusion determinations was carried out using the temperature cycle and reducing furnace atmosphere specified by ASTM. A similar series of tests was carried out using the same temperature cycle but in an oxidizing furnace atmosphere that contained 8% oxygen. Using the ASTM determinations as a basis for comparison it was found that the ash-softening temperature of untreated Dominion coal was as much as 400°F higher under oxidizing furnace conditions, while the ash-fluid temperature was as much as 120°F higher under similar oxidizing conditions. Mixtures of coal and additive followed this trend to varying degrees, and comparative data are given in Table 2. These data illustrate the importance of maintaining oxidizing conditions in fire-beds to improve combustion performance by controlling fluidity or viscosity of ash. This, as explained later, will also influence the nature of clinker formation.

TABLE 2

Influence of Furnace Conditions on Ash-softening and Ash-fluid Temperatures of Dominion 1-1/4 in. x 0 Nut-slack Coal with Selected Additives

Additive	Reducing Atmosphere (ASTM)		Oxidizing Atmosphere (8% O_2)	
	Softening Temp. °F	Fluid Temp. °F	Softening Temp. °F	Fluid Temp. °F
2%CaO	1950	1990	2300	2460
5%CaO	1985	2010	2280	2450
2%Fe_2O_3	2035	2100	2570	2655
5%Fe_2O_3	2115	2160	2620	2730
2%Al_2O_3	2490	2605	2510	2770
5%Al_2O_3	2630	+2750	2760	+2800
2%SiO_2	2100	2440	2440	2620
5%SiO_2	2180	2560	2480	2680
Dominion Coal	2050	2450	2455	2570

EVALUATION OF CLINKERS FROM COMBUSTION-RIG TESTS

General

To correlate ASTM ash-fusion determinations with practical conditions, clinkering tests were carried out in the laboratory combustion rig mentioned previously(1). In these rig tests, 150 lb batches of Dominion 1-1/4 in. x 0 nut-slack coal containing various amounts of selected additives were burned. The clinkers produced were compared with those obtained from untreated Dominion 1-1/4 in. x 0 nut-slack coal. An evaluation of the results follows.

Evaluation of Ash-fusion Determinations

Figure 1 shows that the ASTM ash-softening temperature can be raised by adding the proper quantity of any of the principal oxides found in the ash. It would seem logical that an increase in ash-softening temperature would correspondingly decrease clinker formation. This, unfortunately, was not found to be the case.

An inspection of Figure 1 shows that above 2000°F the same ash-softening temperature can be obtained with two values of ash index. However, it was demonstrated in combustion-rig tests that the clinkering characteristics of such compositions differ widely. A description of the procedure used in these tests follows.

Two 150 lb batches of Dominion 1-1/4 in. x 0 nut-slack coal were prepared, both having additives to provide an ash-softening temperature of 2100°F, but iron oxide was used in one to obtain the lower value of ash index while silica was used in the other to obtain the higher value. These batches were burned in the laboratory combustion rig and, despite their identical ash-softening temperatures, the clinker from the coal containing iron oxide was smooth, thin and dense, while that from the coal containing silica was porous and fluffy. The difference could only be attributed to the viscosity of the ash at the temperature conditions that prevailed in the fire-bed.

This demonstrates that the ash-softening temperature alone is not a reliable basis for anticipating the type of clinker formation because it does not indicate the degree of fluidity of ash in a fire-bed. More important is the ash-fusibility range, which is the difference between the ash-softening and ash-fluid temperatures. If the fusibility range is narrow, a small increase in temperature beyond the softening temperature causes the ash to become fluid; thus clinker forms on cooling. If the ash-fusibility range is wide, a similar increase in temperature results in a viscous ash. Thus, if two coals have similar ash-softening temperatures, the one having a narrower ash-fusibility range is more likely to form troublesome, dense clinker.

From Table 2 it can be seen that the ASTM ash-fusibility range varied from 25°F to 65°F for the coal with lime and iron oxide additives, and from 115°F to 380°F for the coal with silica and alumina additives. Localized fire-bed temperatures in the combustion rig were found to reach 2600°F; therefore, it was anticipated that coal having silica or alumina additions would produce less-fluid ash and would form less-troublesome clinkers than coal having lime or iron oxide additions. This was verified in further tests.

Evaluation of Ash Viscosity

A direct measurement of the fluidity or viscosity of ash entails the use of special high-temperature apparatus and laboratory techniques, but, for practical purposes of understanding the condition of ash in a fire-bed, an indirect evaluation of ash viscosity is adequate, and indeed preferred.

An ash that is fluid in a fire-bed has been known, upon cooling, to produce a dense clinker while a viscous ash has been known to produce a less dense, porous clinker. Thus, the indirect evaluation of ash viscosity consisted of a measurement of clinker density and a relative measurement of the physical strength of the clinker in a drop-shatter test.

The drop-shatter test developed for this investigation consisted of twice dropping a sample of clinker from a height of six feet onto a steel plate. The shattered pieces were then sieved through a series of square-holed screens ranging from 1/2 in. to 2 in., and the percentage weight of clinker retained on each screen was recorded.

Accordingly, all clinkers from the combustion-rig tests were weighed and then subjected to density determinations and drop-shatter tests. As previously mentioned, the clinker from untreated Dominion 1-1/4 in. x 0 nut-slack coal was used as a standard against which that from each additive mixture was compared.

The weight and density of the clinkers produced from burning 150 lb batches of coal with various additives are given in Table 3. The line marked "removal" indicates the difficulty encountered in removing the clinker from the rig. It can be seen that as the clinker became more dense it adhered more strongly to the retort of the combustion rig.

A summary of the effect of additives on Dominion coal is given in Table 3 and results of the drop-shatter tests are plotted in Figures 2 to 9. The table and graphs show that, compared with the untreated coal, silica and alumina additions produced a greater percentage of plus 2 in. fragments, while lime and iron oxide additions produced a smaller percentage of plus 2 in. fragments. Thus it may be concluded that the impact strength of the clinker from Dominion 1-1/4 in. x 0 nut-slack coal is increased by silica and alumina additions, and reduced by lime and iron oxide additions.

The clinkers from fluid ash produced by mixtures containing lime and iron oxide adhered to the retort and were, therefore, difficult to remove from the combustion rig. From this it can be concluded that troublesome clinkers have relatively low impact strengths. Conversely, the clinkers from viscous ash produced by mixtures containing alumina and silica were easily removed from the combustion rig; therefore, acceptable clinkers are characterized by relatively high impact strength. These clinkers are fluffier, thicker, less dense and more resilient than clinker from untreated Dominion coal.

Clinkering tests were also conducted to evaluate a well-known commercial additive which claimed beneficiation of coal combustion properties by reduction of clinker density, elimination of fly ash, reduction of smoke emission and reduction of fuel consumption. The results of these experiments were primarily negative. From the graph of the drop-shatter test, Figure 7, it might appear that the additive caused a slight improvement in the clinkering characteristics of Dominion coal. However, this was not the case, because the graph was based on the percentage weight of clinker retained on each sieve. An inspection of Table 3 will show that the clinker formed from Dominion coal with the commercial additive was heavier, denser and more difficult to remove than that formed from the natural coal. Therefore, this particular additive actually contributed to the formation of a more objectionable type of clinker instead of making it porous and less dense as intended. The effects of other additives may be compared directly by the drop-shatter test results in Table 3, because the respective additives caused little variation in the total weights of clinker compared with that from untreated Dominion coal.

The results of ash viscosity evaluations support practical experience which has shown that dense, fluid clinkers adhere to grates, block air openings and are difficult to remove from stoker grates. In practice, such clinkers are more objectionable than less-fluid, porous clinkers.

TABLE 3

Summary of the Effects of Additives on Dominion 1-1/4 in. x 0 Nu -slack Coal

	3-1/2% SiO_2	2% Al_2O_3	70% Dominion 30% Canmore	2% SiO_2	100% Dominion Coal	1-1/3 % Commercial Additive	2% CaO	3-1/2% CaO	2% Fe_2O_3
Free-swelling Index	8-1/2	8-1/2	4-1/2	8-1/2	8-1/2	8-1/2	8-1/2	8-1/2	5
Clinker Weight, lb	6.32	7.36	6.02	6.66	6.38	9.53	7.46	7.63	6.51
Clinker Density, gm/cm^3	2.18	2.06	1.81	1.91	2.45	2.54	2.51	2.86	2.71
Clinker Drop-shatter Test, + 2 in. Fragments, %	54.2	34.9	24.3	23.6	15.7	16.7	12.5	11.2	11.1
*Ash-softening Temperature, °F	2120	2490	2300	2100	2050	2090	1950	2040	2035
*Ash-fluid Temperature, °F	2380	2605	2570	2440	2450	2280	1990	2110	2100
*Ash-fusibility Range, °F	260	115	270	340	400	190	40	70	65
Ease of Clinker Removal from Rig	Easy	Easy	Easy	Easy	Difficult	Very Difficult	Difficult	Very Difficult	Very Difficult

*These are standard ASTM determinations using a reducing furnace atmosphere.

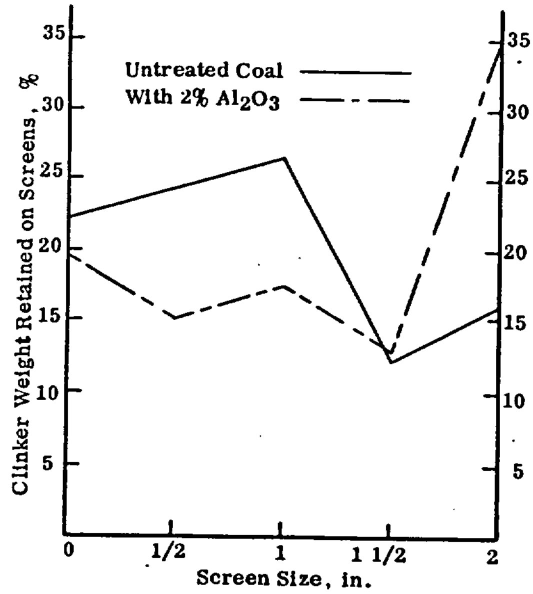


Figure 2. Results of drop-shatter test on clinker from Dominion coal containing 2% Al_2O_3.

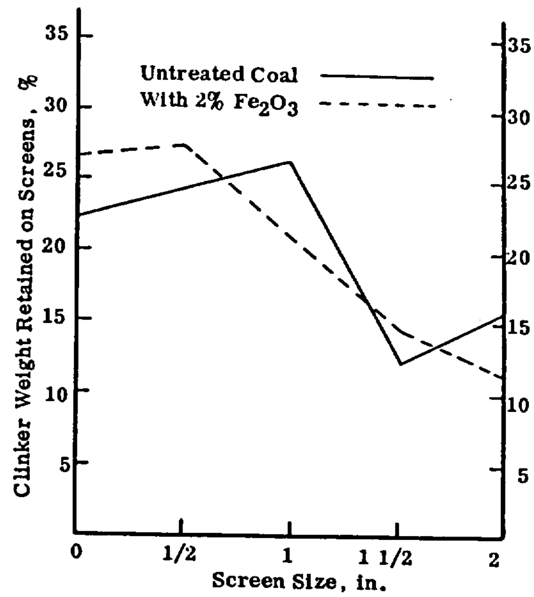


Figure 3. Results of drop-shatter test on clinker from Dominion coal containing 2% Fe_2O_3.

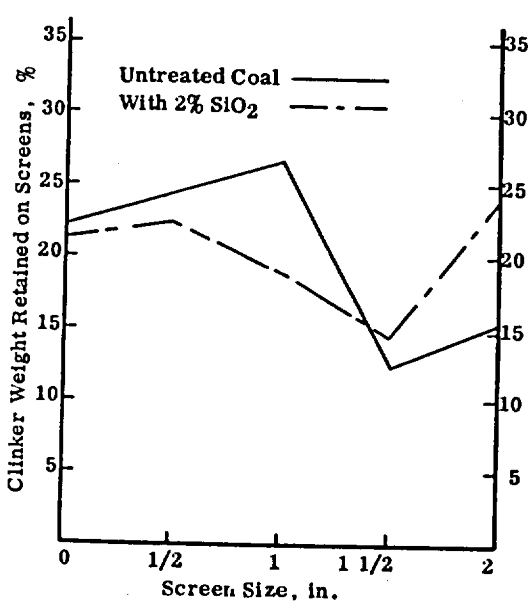


Figure 4. Results of drop-shatter test on clinker from Dominion coal containing 2% SiO_2.

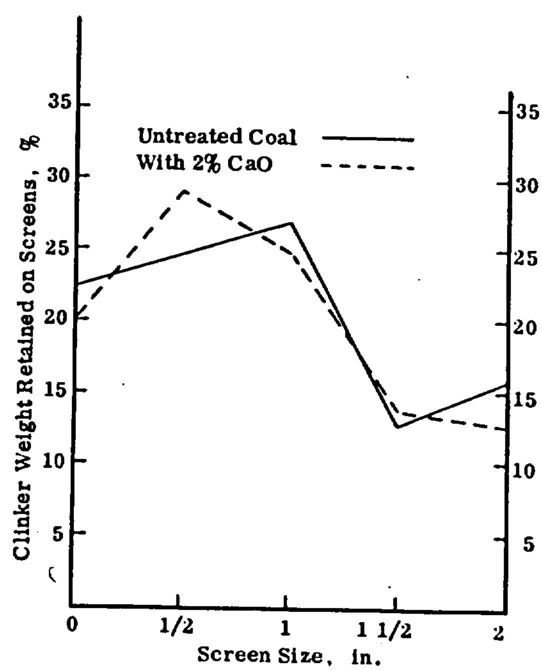


Figure 5. Results of drop-shatter test on clinker from Dominion coal containing 2% CaO.

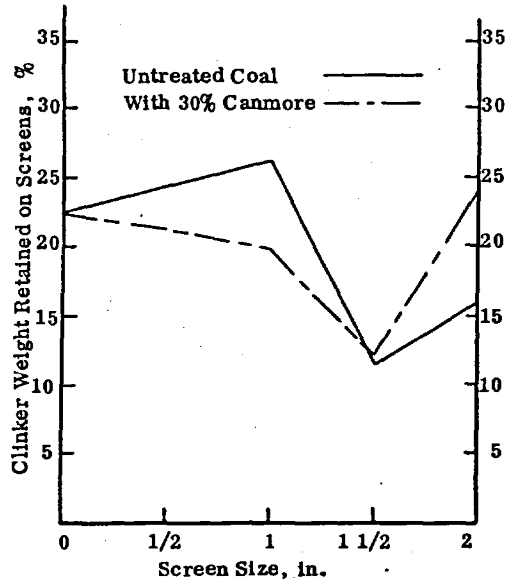


Figure 6. Results of drop-shatter test on clinker from Dominion coal containing 30% Canmore coal.

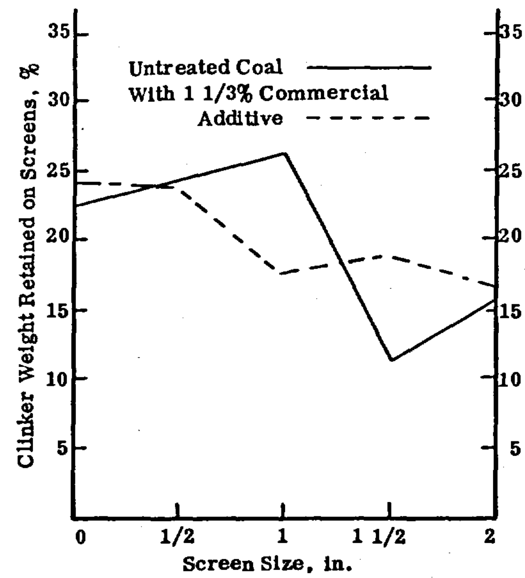


Figure 7. Results of drop-shatter test on clinker from Dominion coal containing 1 1/3% commercial additive.

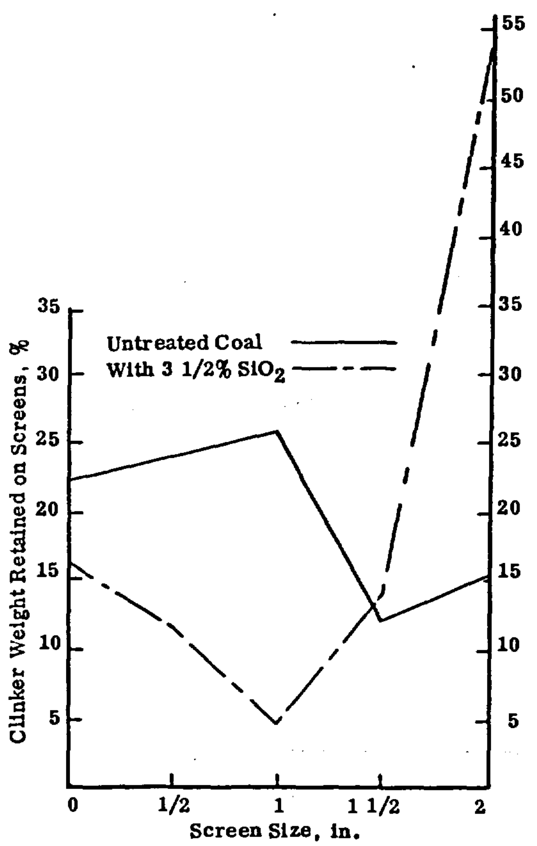


Figure 8. Results of drop-shatter test on clinker from Dominion coal containing 3 1/2% SiO_2.

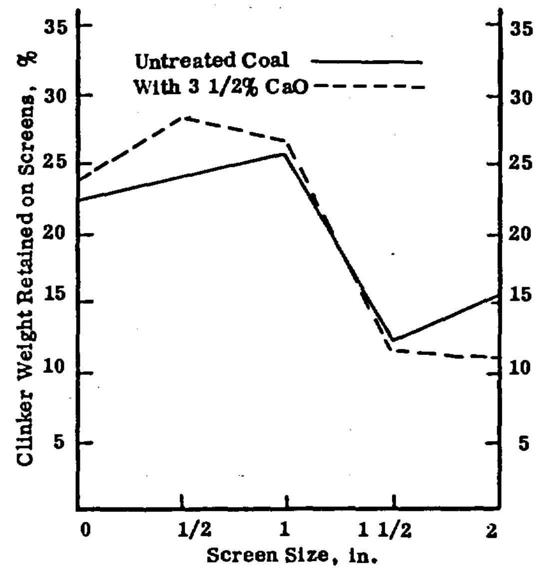


Figure 9. Results of drop-shatter test on clinker from Dominion coal containing 3 1/2% CaO.

PETROGRAPHIC DESCRIPTION OF THREE COMBUSTION-RIG CLINKERS

General

In order to gain a better understanding of the mechanism of clinker formation, the clinkers produced from untreated coal and from coal treated with 2% lime and with 2% silica, were subjected to petrographic examination and X-ray diffraction analysis.

External Surface Features

A stereomicroscopic examination of the external surfaces of the aforementioned three clinkers showed them all to be similar and predominantly grey-black with rust-coloured overtones and with white inclusions randomly embedded. The grey-black surfaces were relatively hard, whereas the white inclusions were friable. An X-ray diffraction analysis of the three visible surface components of one clinker sample is given in Table 4.

TABLE 4

X-ray Diffraction Analysis of Surface Components of Clinker

Component Colour	Composition	Amount Present
Grey-black	Magnetite (Fe_3O_4)	Major
Rust	Hematite (Fe_2O_3)	Minor
White	Mullite ($3\ Al_2O_3 . 2\ SiO_2$)	Minor

Although the surfaces of all three clinkers were similar in composition, their contours were markedly different. The surface of the clinker from the silica mixture was more heavily and deeply cratered than the clinker from untreated coal, while the clinker from the lime mixture contained only a few isolated shallow pits.

Thin-section Investigation

The physical compositions of the three clinkers were determined from a study of thin sections taken through the clinker. This revealed that they all consisted of three common phases, namely: glass, crystalline matter, and voids. Table 5 gives the approximate phase compositions of the thin sections.

TABLE 5

Approximate Phase Composition of Clinker Thin Sections

Phase	Clinker from Lime-treated Dominion Coal	Clinker from Untreated Dominion Coal	Clinker from Silica-treated Dominion Coal
Glass, %	17	19	23
Crystalline Matter, %	78	58	49
Voids, %	5	23	28

The crystalline matter was identified, where possible, by examination under a petrographic microscope and by X-ray diffraction analysis. Table 6 gives the approximate mineral compositions and grain sizes.

Since it has already been established in this report that the fluidity of the clinker from Dominion coal is increased by the addition of lime and reduced by the addition of silica, the data compiled in Tables 5 and 6, although limited to specific thin sections, may indicate the following trends:

1. The clinker from fluid ash is characterized by a large percentage of crystalline matter and a low percentage of voids. Furthermore, a highly fluid ash will favour

formation of crystals in the resulting clinker.

2. On the other hand, viscous ash will form clinker containing more voids and less crystalline matter than fluid ash.

The foregoing data suggest that the relatively low impact strength of clinker produced with lime additions results from the high percentage of crystalline matter and low percentage of voids, while the relatively high impact strength of clinker produced with silica additions results from the low percentage of crystalline matter and high percentage of voids. It seems likely that voids increase impact strength, because the thin walls forming them crush on impact, thus absorbing the shock that would otherwise shatter the clinker.

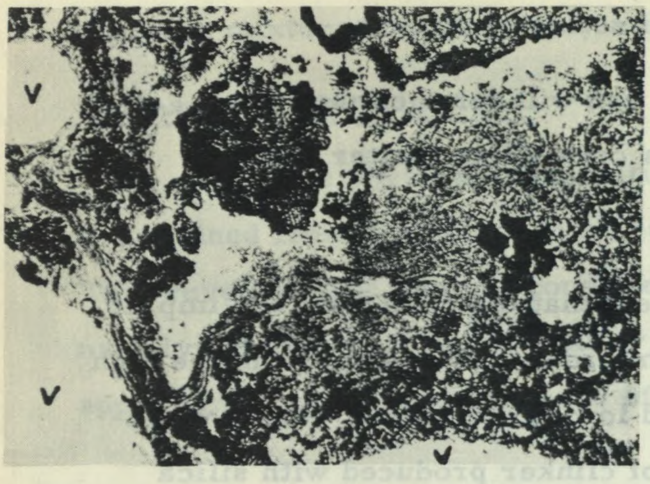
TABLE 6

Approximate Mineral Compositions and Grain Sizes of Crystalline Matter in Specific Clinker Thin Sections

Mineral	Clinker from Lime-treated Dominion Coal		Clinker from Untreated Dominion Coal		Clinker from Silica-treated Dominion Coal	
	Amount Present, %	Grain Size, mm	Amount Present, %	Grain Size, mm	Amount Present, %	Grain Size, mm
Magnetite + Opaque Matter	16	0.001-0.01	99	0.01-1.0	85	0.01-10
Quartz	-	-	1/2	+0.1	14	0.1 -0.5
Unknown	84	0.005-5.0	1/2	0.001-0.005	1	0.0 -0.05

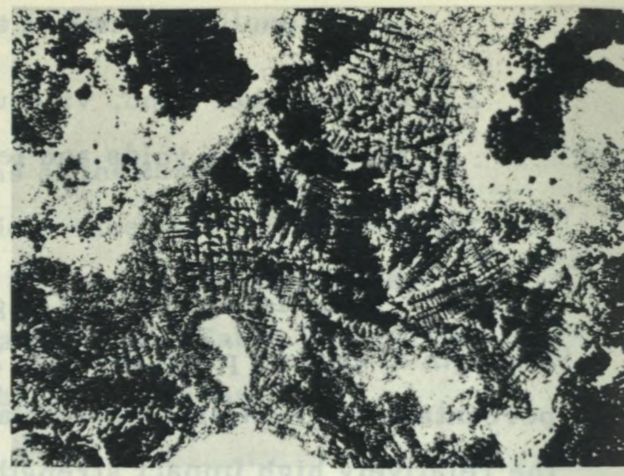
Photomicrographs of Thin Sections

Photomicrographs of the thin sections are shown in Figures 10 to 14. They illustrate areas that are not necessarily typical but selected to show different phases. The white areas



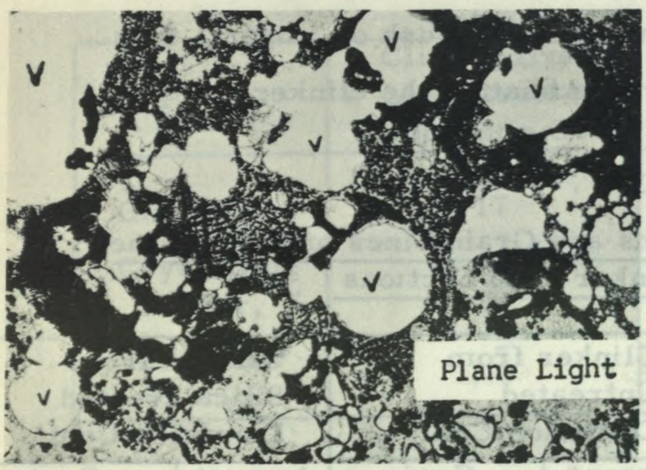
Thin Section x 20

Figure 10. Clinker from untreated Dominion coal.



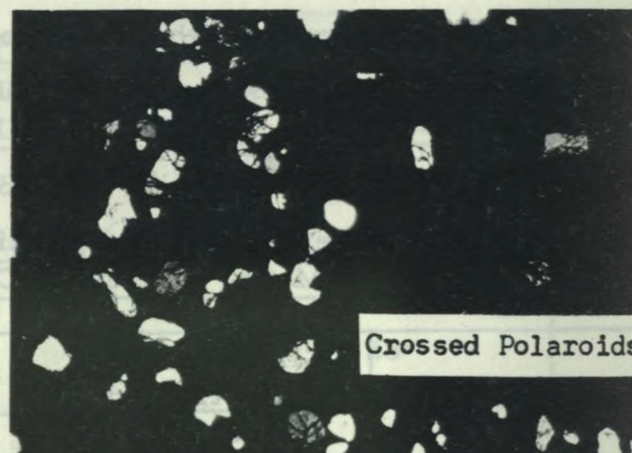
Thin Section x 40

Figure 11. Clinker from untreated Dominion coal.



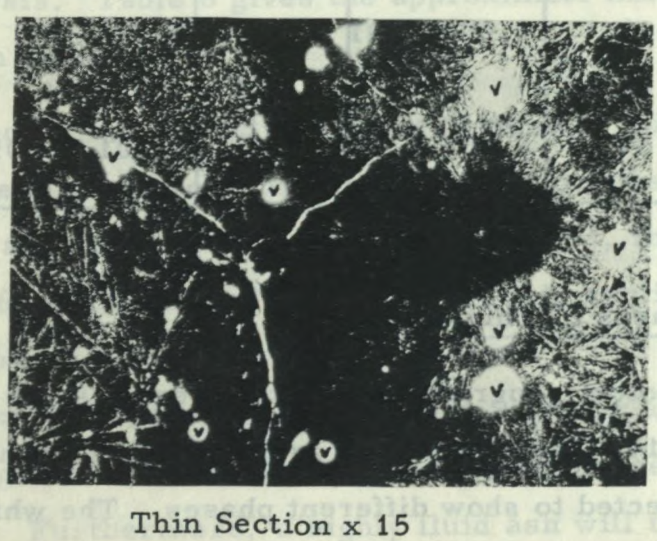
Thin Section x 20

Figure 12. Clinker from silica-treated Dominion coal.



Thin Section x 20

Figure 13. Clinker from silica-treated Dominion Coal.



Thin Section x 15

Figure 14. Clinker from lime-treated Dominion coal.

marked "V" in Figures 10. 12 and 14 are voids. In Figures 10 and 11, relating to clinker from untreated Dominion coal, the black areas are rectilinear networks and nodular clusters of magnetite, while the white areas are an amber glass phase.

EXPERIMENTS WITH CANMORE-DOMINION BLENDS

In searching for additives to improve the burning properties of Dominion coal it was realized that blending with another coal would have the advantage over inert additives of maintaining or increasing the calorific value of Dominion coal without increasing its ash content.

In the past, coal blends have not always been successful. Ashes of different coals in combination sometimes have a lower ash-softening temperature than either of the individual ashes due to thermo-chemical reaction. The preferred ash is one that contains mostly alumina or silica or both. Thus a western Canadian coal named "Canmore" appeared to be suitable for blending since its ash contains some 85% silica and alumina. The compatibility of Canmore with Dominion coal was borne out in ASTM ash-fusion determinations.

From these tests it became apparent that mixes of 25% to 50% Canmore 9/16 in x 5/16 in. buckwheat with Dominion 1-1/4 in. x 0 nut-slack would achieve the desired result and this was demonstrated in extensive combustion-rig tests (Table 3).

Following this, full-scale combustion tests with a blend of 30% Canmore - 70% Dominion were conducted in two Ottawa heating plants equipped with small underfeed stokers and troubled by excessive smoke emission. In both cases, operation was greatly improved with the coal blend because the reduced caking of the coal permitted smoke-free combustion at maximum boiler ratings.

Further experiments in large stokers disclosed that the blend should be limited to use in stokers with retorts no longer than 7 ft. In stokers larger than this the Canmore coal, due to its friable nature, suffers degradation to the extent that the coal at the rear of the retort becomes excessively fine and upsets combustion conditions.

CONCLUSIONS

Although the ASTM ash-fusion determinations and the combustion-rig clinker tests were made under certain fixed conditions that are seldom duplicated in an operational fire-bed, the results provide trends which can be related through experience to actual combustion conditions on stoker grates. The laboratory tests have shown that certain additives, such as silica or a blend with a suitable coal, such as Canmore, can alleviate the more serious caking and clinkering conditions. But, in the light of present knowledge, additives or blends should only be considered if it is not feasible to modify the existing combustion equipment to suit the burning characteristics of Dominion coal. The necessary design features of combustion equipment are described elsewhere(2).

ACKNOWLEDGMENTS

Special credit is due to Dr. J. A. Soles of the Mineral Processing Division for providing the petrographic data. The assistance of J. Custeau, chemist, Fuels and Mining Practice Division, is also gratefully acknowledged.

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