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BRIQUETTING COAL WITH BINDERS AND STATISTICAL EVALUATION OF BRIQUET TESTS

by

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BRIQUETTING COAL WITH BINDERS AND STATISTICAL EVALUATION OF BRIQUET TESTS

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R.P. Charbonnier* and J. Visman**

ABSTRACT

In view of the difficulties of controlling the various factors of coal briquetting with binder, statistical methods are very useful in the investigations of the briquetting process in laboratories, and even more so in industrial plants.

A summary of the most important recent developments in coal briquetting with binders is first given, in order to help in the planning of specific research programs under local conditions of individual briquetting plants.

After a summary of previous studies on the evaluation of the physical quality of briquets, an application of certain statistical methods is described, including factorial designs of experiments; analyses of variance; tests of significance; and correlation, regression and index formulae. A detailed description of such techniques is given with a view to facilitating the organization and analysis of research programs by agglomeration engineers. For the convenience of the reader, the statistical data are gathered in an Appendix.

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AGGLOMÉRATION DU CHARBON AVEC LIANTS ET ANALYSE STATISTIQUE D'ESSAIS D'AGGLOMÉRATION

par

R.P. Charbonnier* et J. Visman**

RÉSUMÉ

Les divers facteurs de l'agglomération de la houille avec liants étant difficiles à contrôler, il est très utile de faire appel aux mathématiques statistiques pour analyser les résultats d'essais d'agglomération sous diverses conditions, au laboratoire et--à fortiori--dans les briquetteries (usines d'agglomération).

Ce mémoire commence par une étude d'ensemble des plus importantes contributions récentes à la technique de l'agglomération du charbon avec liants en vue de faciliter l'élaboration des programmes de recherche dans les cas particuliers donnés par les conditions locales de chaque briquetterie.

Après un rappel de recherches antérieures sur les essais de qualité mécanique des agglomérés, les auteurs décrivent une application de certaines méthodes statistiques, telles que les plans factoriels d'expériences, l'analyse de variance, les tests de signification, et les formules de corrélation, de régression et d'index. Une description détaillée de ces méthodes est donnée afin d'aider les ingénieurs de briquetteries à analyser leurs recherches sur l'agglomération. Pour simplifier l'exposé, les résultats statistiques sont rassemblés en un Appendice.

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INTRODUCTION

Agglomeration, and in particular briquetting, is a very important process in the mining, metallurgical and chemical industries. Recent researches in coal briquetting with binders are surveyed in the present report, with the purpose of helping preparation engineers to conduct tests in their own plants with a view to improving the quality and reducing the cost of their briquetting operations.

In Section I, a brief summary of the most salient points of recent research developments in the briquetting of coal with binder will be given. Although many engineers, who are not concerned with cost reduction and improvement of quality, usually regard briquetting as simple, it is in fact a complicated process because of the numerous factors which are important either by themselves or by their interactions and which are difficult and expensive to control adequately.

Research investigations on the influence of the various factors of briquetting have endeavoured to reduce the costs of production and improve the quality of the briquets. This quality is usually evaluated by measurements of certain physical properties, such as resistance to compression or bending ("strength" index) and resistance to shattering and/or abrasion ("stability" index), and by combustion tests, strength measurements after devolatilization, weathering tests,and chemical characteristics. Herein, only the physical properties of the briquets are considered. Unfortunately, no standards of testing procedures have yet been established.

Section II of this paper will deal first with the evaluation of the physical quality of briquets by two simple tests, namely the compressive strength test and the tumbler test, both of which are reliable under certain conditions which are most practical from the plant operator's viewpoint and studies of which have appeared in previous publications.(4,5,6)

After suitable procedures for evaluating the physical quality of briquets have been described, some new testing methods will then be examined. These were introduced early in 1954 in briquetting plants in Canada to evaluate, by statistical techniques, the respective influence of the most important factors of briquetting.

In this report, the general principles and practices of coal briquetting are assumed to be known and therefore no details of general knowledge are given in view of the abundant literature available on the subject (see, for example, references 1 and 2).

I - INFLUENCE OF THE MOST IMPORTANT FACTORS OF COAL BRIQUETTING WITH BINDERS

Only the most outstanding recent technological improvements will be discussed, each influencing factor being examined separately: nature and size of the coal, binders (coal tar pitch, petroleum asphalts, starch, and sulphite liquor), moisture, and temperatures and briquetting technique.

Coal

Nature of the Coal

For proper combustion behaviour of the briquets, the coal must have suitable coking properties, otherwise the briquets may require expensive treatment (such as partial carbonization). When the coking properties are deficient, as is usually the case in certain types of subbituminous and anthracite coals, this difficulty may be solved by the admixture of ten to twenty percent of suitable coking coal to the coal to be briquetted.

High porosity and moisture of the coal may be detrimental to briquetting by increasing the binder requirements. Generally, up to the semianthracites, the higher the rank of the coal the better the physical properties of the briquets.

Although the nature of the coal is a very important factor, especially as to the chemical and physical characteristics of the briquets, usually it can hardly be controlled or modified by the briquetting plant operator.

Size of the Coal

The optimum size consist of the coal is that giving maximum packing density and minimum crushing in the mixer, fluxer and press, and seems to be close to a normal distribution in size curves (plotted on probability-logarithm paper as a straight line in cumulative percentages by weight versus size openings in mm). Usually, this sizing can easily be approximated by hammer mill crushing of coarse coal, provided that no particular size range is increased or decreased in percentage by adding other coals or by screening out some sizes.

Recent investigations on typical bituminous coals showed that, in general, the compressive strength of briquets increased when the top size of the coal particles was raised up to 1/8 in., remained constant when the top size was increased from 1/8 in. to 1/4 in., and decreased when the top size exceeded 1/4 in.

It is often assumed that an increase in the proportion of small sizes and coal dust in the briquetting mix makes necessary, if the mechanical quality of the briquets is not to be reduced, a corresponding increase in the amount of binder. However, there is more and more evidence that the above assumption is not correct. For instance, in recent studies the proportion of minus 50 mesh coal was increased from 36 to 52 percent, the 35 x 50 mesh from 18 to 26 percent and the 18 x 35 mesh from 15 to 22 percent, with little or no change in the mechanical characteristics of the briquets. Likewise, very fine flotation products (up to 74 percent of minus 50 mesh) have been mixed with the usual size of briquetting coal in all proportions without significant detrimental effects. The specific surfaces varied from 67 to 292 cm^2/gram. In some cases it has been found that the elimination of plus 10 mesh coal improves the briquet. The relative proportions of the various size fractions of the coal appear to be more important than the amount of fine coal or dust. For a typical bituminous coal the optimum size distribution was found to be as follows: 1/8 in. x 18 mesh, 50 percent; 18 x 50 mesh, 30 percent; minus 50 mesh, 20 percent. These results are supported by microscope examinations of briquets, which have shown that the asphalt binder makes a sort of "concrete" with very fine coal particles and that the large coal particles are held together by "bridges" of this "concrete", entrapping the smaller particles and leaving many cavities in between.

Binders

Coal Tar Pitch

Coal tar pitch is still the binder most extensively used in the world - especially in Europe, where there seems to be a definite trend toward the use of molten, rather than crushed, pitch because of the easier handling and other advantages of softer pitches with high adhesive power. Coal tar pitch may be softened by the addition of about 15 percent of anthracene oil or tar oil, with a resulting reduction from about 72°C to 45°C in the Kraemer-Sarnow softening point*. With such softer pitches, binder consumption may be reduced by about 1.5 percent, as one part of oil replaces two parts of pitch (Martel Process).

Coal tar oils, including anthracene, used in the Convertol process for dewatering fine washed coal prior to briquetting, result in a reduction of binder requirements.

When using crushed coal tar pitch, the optimum top size of the pitch particles appears to be approximately 14 mesh, with an average particle size of 120 mesh.

Petroleum Asphalts

Generally, petroleum asphalts, either of paraffinic or of asphaltic origin, are better binders than coal tar pitch but show lower temperature susceptibility and therefore require more cooling of the briquets after pressing. They also show poorer coking properties.

Just as each coal presents a different briquetting problem, each asphalt shows a different binding ability by itself, and also according to the coal briquetted with it. Aromatic (cyclic) compounds usually give better adhesion than do aliphatic (open-chain) compounds, even with the more mature and hydrophobic coals.

For typical Canadian bituminous coals (7, 8) the compressive strength of the briquets may increase by 25 to 35 percent of its

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*The Kraemer-Sarnow softening point values are, on the average, about 10°C lower than the ASTM Ring and Ball values.

value, and the stability index by 1 to 15 percent (absolute), for every increase of one percent in asphalt content. Generally speaking, and notwithstanding many exceptions, cracking coil asphalts have briquetting properties nearest to coal tar pitch. "Straight-run" asphalts obtained from direct distillation often give briquets with better compressive strength but lower temperature susceptibility and slightly lower resistance to abrasion.

In most cases, when testing briquets at ordinary room temperatures, their stability index increases when the penetration of the asphalt increases and, with a much lower degree of correlation, when the viscosity of the asphalt (at 350°C to 400°F) increases. Compressive strength usually increases as the penetration index of the asphalt decreases.

Optimum values of the various characteristics of the asphalt binder for any briquetting operation should be obtained by compromise according to the local conditions ascertained by experimenting. If the asphalt is added to the mix in a ground and cold state, the optimum Kraemer-Sarnow softening point of the asphalt varies between 50 and 85°C, according to the nature and method of processing of the crudes, the briquetting techniques, the nature of the coals, and the outside temperature. If the asphalt is added in the molten state, its softening point should be about 10°C lower. Optimum penetration, defined by the ASTM test: 100 grams, 5 seconds at 25°C (77°F), may vary between 5 and 35, usually 10 to 20.

Improvement of the physical properties of the briquets may be obtained by a better distribution of the binder, resulting from the fine spraying of the asphalt, either by steam or by preheated compressed air or gases, with a nozzle such as the cyclone atomizer. The air would tend to "blow" the asphalt, thus raising the softening point. Experiments have indicated that by atomizing the asphalt an increase in compressive strength equivalent to 0.5% of binder can be obtained.(3)

The comparison of viscosity characteristics of various asphalts is made easier by representing their variations as functions of the temperature on a log-log scale, which gives straight lines. Likewise, the study of the penetration indices of various asphalts is facilitated by representing their variations as functions of the temperature on a log-normal scale, which gives straight lines with slopes corresponding to the temperature susceptibility relationship. For briquetting purposes, the higher the temperature susceptibility of the binder the better the physical quality of the briquets at press, storage and loading temperatures, thereby permitting low degradation losses.

In connection with the combustion quality of briquets, it seems that the cracking coil asphalts often have coking properties almost as good as those of the coal tar pitches and significantly better than those of the vacuum distillation asphalts or even the slightly better "deasphalting" asphalts produced by propane precipitation. The agglomerating power of an asphalt generally improves when its softening point is lowered. Briquets made with asphalt are usually stronger and less affected by excessive moisture than are those made with coal tar pitches.

For use in the molten state, it seems that the optimum specifications for an asphalt binder used in briquetting typical bituminous coals are as follows:

Origin: cracking coil

Ring and Ball softening point: 65-70°C (150-160°F)

Penetration: maximum at 46°C (115°F): 35

" : minimum at 66°C (150°F): 120

" : optimum at 25°C (77°F): 5-20

Susceptibility: ratio penetration at 66°C/penetration at 46°C: minimum: 4.5

Moisture: maximum 0.5%

Ash content: maximum 0.5%

Insoluble in benzene: maximum 1%

Conradson index: minimum 35

Starch and Sulphite Liquor

As a binder, starch in aqueous solutions (1 part starch with 5 to 10 parts water) has often proved about twice as effective by weight (on a dry basis) as asphalt, especially when briquetting immature coals. Starch should be used as part of a dual binder with one or two percent asphalt, the latter to provide sufficient resistance to weathering.(7,8)

Similarly, sulphite liquor may also be used in a dual binder with about 3 percent asphalt (for weathering protection), but the temperature susceptibility of the briquets is low and storage is also usually necessary for the long curing time required. Drying or partial carbonization of these briquets may often be necessary.

Moisture

Surface moisture is one of the most important factors in coal briquetting with a binder, but inherent moisture is generally regarded as negligible as to its influence on the agglomeration process.

Whereas the compressive strength of briquets bound with crushed coal tar pitch is generally maximum for a water content of the briquetted mix between 2 and 4.5 percent, there does not seem to be an optimum when binding either with molten coal tar pitch or with petroleum asphalts. Then any moisture appears to be detrimental - more or less according to the nature and size consist of the coal, the temperatures used, the binder, etc. Although the effects of sudden increases of the moisture content may be compensated by raising the binder percentage, this solution is costly and difficult to apply because the moisture content is not easy to evaluate quickly and to control adequately. Recent investigations lead to the generalized use of controlled heat drying of the coal, which should then be introduced dry in the mixer at about 90°C. With such a hot coal, steam injections in the mixer and the fluxer would not result in the considerable condensations of water that are often observed (5 to 8% water thus added to the mix in many plants). Many operators now inject superheated steam (up to 350°C) in large amounts to raise the temperature of the mix by specific heat rather than by the latent heat of evaporation which results in considerable condensation. The injection of hot inert combustion gases has also been advocated.

For typical Canadian bituminous coals, the compressive strength of the briquets may decrease by 10 to 25 percent of its value, and the stability index by 2½ points, for every increase of one percent in moisture content of the mix to the press.(7,8)

Temperatures and Briquetting Technique

Adequate control of the various temperatures in the briquetting process is very important.

Recent investigations have shown that the following conditions may be optimum for typical bituminous coals: the coal tar pitch or petroleum asphalt may be sprayed at approximately 200°C (when its viscosity is between 10 and 80 centistokes) by mechanical pressure (about 12 kg/cm^2) or by dry steam or hot compressed air (above 3 kg/cm^2) against a vertical curtain (about one foot wide and one inch thick) of coal, preheated to about 90°C and dried to approximately 2% moisture.

Mixing and vertical fluxing during 7 to 10 minutes should raise the temperature to about 100°C. Then the mix should be cooled for pressing to a temperature about 5 to 10°C above the Kraemer-Sarnow softening point of the binder.

Cooling the mix is generally difficult. The best results may be obtained by using a reverse draft of dry cold air over the mix while it is conveyed in a screw or paddle conveyor and a flight conveyor equipped with compressed air injectors. This would improve the cooling obtained by vaporization of excessive moisture in the mix.

It would be desirable to increase the pressing time and pressure beyond the 1/10 to 2/10 of a second and approximately 150 to 200 kg/cm^2 (2000-3000 psi) usual for roll presses, for these conditions may not allow sufficient evacuation of the air and steam contained in the mix before pressing. The density of the mix is about 0.7, which must be raised to about 1.2 or 1.3 in the briquet by pressing. Ring-roll presses facilitate the evacuation of the gases from the mix and may increase the pressure to about 1500 kg/cm^2 (22,000 psi). Open mould plunger presses may increase the time of pressing to 5 to 10 seconds and the pressure to 400 kg/cm^2 (6000 psi) or more. Excellent results have been obtained with both these types of equipment, using only 3.5 percent of coal tar pitch instead of the 7 percent required to get the same quality of briquets with roll presses, but their application is not yet possible, because of the high maintenance and/or capital costs involved. Larger roll diameters (5 feet or more) and roll presses with forced feeding have also been advocated with a view to increasing the pressure.

Vibrating the mix in the press feed box has been considered as a means of facilitating the evacuation of the gases from the mix prior to pressing, but preliminary tests have not been successful.

Cooling the briquets before they are loaded is essential; however, it is not often done effectively. For briquets bound with coal tar pitch, cooling times vary from 2 to 20 minutes with 1 to 5 layers of briquets on the conveyor. Under these conditions their temperature may be 70°C (158°F) when being loaded. Lower loading temperatures are desirable, especially when using asphalt as the binder, and the briquets should have an average temperature about 40°C (70°F) below the Ring and Ball softening point of the asphalt (that is, approximately 30°C or 90°F) when they are loaded or stored. Such cooling may be obtained by screening out the broken fins and degradation material as early as possible after pressing, and by repeated slight water spraying and/or cold air draft over the briquets on the cooling conveyor.

II APPLICATION OF STATISTICAL METHODS TO THE STUDY OF BRIQUETTING UNDER PLANT CONDITIONS

Evaluation of the Physical Quality of Asphalt-Bonded Coal Briquets

Compression Strength

Generally, in plant practice or in laboratory investigations, the physical characteristics of coal briquets are judged by determination of their resistance to crushing. Therefore, it was necessary to first ascertain under what conditions the compression strength test, usually applied in North America, was reliable. This study has been reported elsewhere(4, 5) and only the conclusions are summarized below:

Under controlled conditions, the Komarek-Greaves compression strength test is reliable and can be used as a dependable yardstick for evaluating the physical quality of pillow-shaped roll press briquets made of bituminous coal and petroleum asphalt.

The suitable conditions must be determined in each case. The temperatures of the briquets while they are tested should be close to the average temperature to which they may be exposed during transportation and storage. Corrections for temperature must be made in the order of one Komarek-Greaves (K-G) unit per degree Fahrenheit. The exact corrections required vary according to the type of briquets.

The speed of the tester should be approximately 200 rpm in order to ensure reproducible results.

If obtained at the same speed and corrected to the same temperature, results can be compared for different samples of briquets with good precision (in the order of 3 K-G units for 5 replicates). Greater precision can be obtained by using a larger number of replicate briquets.

Within the range of ordinary room temperatures, the accuracy(4,5) of the K-G test, evaluated by correlation with the instantaneous FRL test of the Canadian Department of Mines and Technical Surveys, is acceptable at high speeds in the order of 200 rpm.

Shattering and Abrasion Stability

Another physical property of briquets which is very important and often used for comparison of various samples of briquets is the resistance to shattering and abrasion. Therefore, a special study was made to determine under what conditions the ASTM D441-45 "Tumbler Test for Coal", as often applied to briquets in North America, was reliable. This investigation of the Tumbler test has already been reported in previous publications(4, 6) and, therefore, only the conclusions will be summarized here, as follows:

The ASTM D441-45 Tumbler Test was found to be reliable for asphalt-bonded bituminous coal briquets weighing approximately three ounces, under the following optimum conditions:

Speed of rotation: 40 to 60 rpm
Number of briquets in each jar: in the order of 12
Duration of tumbling: 15 to 20 minutes
Temperature: about 20°C

Briquet "stability" increases with the temperature of testing in the range 0 - 35°C, decreases with the duration of tumbling (almost linearly between 15 minutes and 3 hours), is minimum for a tumbling speed about 60 rpm, and increases with increasing loads of briquets in the tumbling jar. The stability index may be defined as the percentage of the original weight retained on a one-inch square-mesh screen after 15 minutes at an arbitrary constant temperature about 20°C. If the temperature, the duration, the load and/or the speed of tumbling are different when comparing the qualities of different briquets, corrections should be made assuming that the variations of stability due to these factors are linear within a limited range. In particular, the results should be corrected to an arbitrary temperature close to the average temperature which the briquets may be expected to have during transportation. Alternatively, the tumbler test can be characterized by the rate of degradation of the briquets after 15 to 20 minutes of tumbling. Under these conditions, with the briquets tested in this investigation, three replicate tests are sufficient to obtain an adequate precision in the order of three units of the stability index.

The physical quality of the briquets prepared in the tests referred to in this study has been evaluated according to the determinations of their compressive strength and tumbler stability in the light of the findings summarized above.

The experimental techniques which were used will now be described.

Statistical Methods

Laboratory tests are usually conducted on the basis of controlled variations of a single factor while trying to keep all other factors and conditions as nearly constant as possible. In coal briquetting, this method is difficult to apply because of the many factors involved, their variability and their interactions. When experimenting in a plant instead of a laboratory, such a controlled investigation is practically impossible. As it is difficult even in a laboratory, and also because laboratory test results can seldom be directly applied to industrial practice, it is often desirable that experiments be conducted in the plants under actual operating conditions. Then, the uncontrolled variation of conditions throughout the plant makes it necessary to carry out considerably more tests than in laboratories.

Their programming and analysis are most satisfactorily undertaken by statistical methods, such as factorial design of experiments, analysis of variance, calculations of regression and correlation formulae, and applications of tests of significance such as F-tests (Snedecor) and t-tests (Student-Fisher). The minimum number of tests required is greatly reduced by the use of these statistical methods.(7,8) The influence of each factor, and of the various interactions of factors, can be ascertained and quantitatively expressed relatively to the errors of sampling of the briquets, the precision of the tests performed on the briquets, and the effect of any other factor unaccounted for.

Factorial Plan

Without going into too much detail, the essence of a simple two-levels factorial test design to study the respective influences of, say, four factors of a process may be described as follows:

The effect of a certain factor, e.g. asphalt content, is determined by doing a series of sixteen tests, half of which are made at low asphalt content and the other half at relatively high asphalt content. The "low" tests are kept at the same low level as much as possible and a similar uniform "high" level is aimed at for the "high" tests. The two series are then compared and the differences in strength (or tumbler stability) can be ascribed to the difference in asphalt content if the influence of the other factors can be assumed to cancel out for each series of tests.

Ordinarily, the same procedure would have to be followed for the three other factors, resulting in a total of sixty-four tests in order to obtain, for the three other factors, the same degree of reliability as for the first factor. The "factorial test" plan, however, provides a means of obtaining an evaluation of the influence of all the various factors with the same accuracy as above from only sixteen tests, by a judicious combination of the levels of the factors in each test and by the analysis of the partial variances of the results, corresponding to the various factors and to their interactions.

By averaging the results of the eight "high" tests or the eight "low" tests for one of the factors, e.g. asphalt content, the effects of the changes in the other factors can be practically cancelled out because of a symmetrical arrangement of the "high" and "low" values of the other factors. Similarly, after re-arranging the sixteen tests in two groups of eight "high" and eight "low" values of the second factor - say moisture content - the effects of the asphalt content and of the two other factors are nearly nullified. This procedure of "nullifying" the concomitant factors with the exception of one is not perfect, since the various "levels" of the factors cannot be perfectly controlled in most cases; but generally the effect of a single factor at two levels is much larger than the effects of the other three factors combined and "averaged" or nullified at each of the two levels.

A statistical "test of significance" is done in each case to ascertain this point.

Example

In order to illustrate this technique, an example of briquetting tests will now be described.

A series of tests was carried out at a large briquetting plant in Western Canada to investigate the influence of various factors of coal briquetting with binder. The coal was ranked as medium volatile bituminous (ASTM Classification). From each of the samples of briquets obtained during the tests, ten briquets were taken at random and tested on the K-G Compressive Strength tester and the results were corrected to a curing time of one hour and a testing temperature of 70°F.

Samples of approximately 1,000 grams were tested with a modification of the ASTM Tumbler Test for coal D441-45. Tumbling speed was 40 rpm. The stability index was defined as the average of the percentages of material retained on a one-inch square-mesh screen after 15 and 20 minutes of tumbling, and the abradability index was defined as the average of the percentages of material passing through a 10-mesh Tyler screen after 15 and 20 minutes. The sum of these two indices is usually very close to 100 in the case of bituminous coal briquets.

The following factors of briquetting were studied:

(a) binder: asphalt percentage in the range of 2.9 to 5.0%;

(b) " : type of asphalt (straight run distillation or cracking);

(c) binder: flour percentage in the range of 0 to 1.5%;

(d) moisture percentage in the range of 3 to 8%;

(e) degree of dispersion of the asphalt in the mix (emulsifier or atomizer nozzle).

All the other conditions of the briquetting tests were kept as constant as possible and recorded. They were typical of the briquetting of bituminous coal with binder in Canada.

A number of summaries of selected test results obtained are given in the Appendix, together with some of the elements of the factorial designs, analyses of variances, regression formulae and several statistical tests used in this study.

It must be borne in mind that the following conclusions are valid only within the range of conditions considered in each series of tests, not only in respect to asphalt, flour and moisture contents, etc., but also for the various temperatures at the different stages of the process and all other plant conditions, such as size of coal, equipment characteristics, etc. For instance, it may be that if the temperature of the preheater or mixer were higher (say 205°F instead of the actually recorded 175 to 190°F), the flour binder would be more active, since in many plants and laboratories the flour solution is heated to the boiling point in order to obtain maximum binding properties. It should be noted that the temperature of the fluxer was 195 to 200°F and that of the press was 170 to 175°F.

The conclusions reached after computing statistical analyses of the type summarized in the Appendix are presented here in the form of simple diagrams, which schematize the respective effects of the various factors under study. The variations in physical quality of the briquets are represented here by straight lines which are determined by two points: each corresponding to the average of four tests respectively at high and low levels of the particular factor considered. It must be emphasized that these lines simply represent trends and should not be taken as illustrations of strictly linear relationships. Corrections have been made in each case for the influence of the other factors (e.g. moisture or asphalt content) in order that the comparison of each set of two averages be undistorted by the differences in the other factors, which could not be kept under sufficient control.

Three ranges of variations of the factors were considered in this investigation.

The first range of conditions of briquetting was as follows: 2.9 - 3.6% of asphalt, 1 - 1.5% of flour, and 5.0 to 8.3% of moisture content.

Figures 1, 2 and 3 show the average variations in compressive strength and abrasion stability of the briquets in function of the percentages of asphalt, flour and moisture in that range.

Generally, the range chosen for each factor depends on the importance of the factor, the most preponderant being studied within the narrowest range suitable.

The average level of each range depends on the typical conditions found in practice (e.g. 5% moisture content) and economic conditions (e.g., with dual binders, a relatively low and cheap level of asphalt - say 3% - may be associated with a relatively high and expensive level of flour - say 1.5%).

The second range of conditions studied here covered higher asphalt contents and therefore lower flour contents since commercially the combined cost of binder should not be excessive. This range was: 3.6 - 4.3% of asphalt, 0 - 1.0% of flour, and 3.1 - 5.8% of moisture content.

Fig. 1.

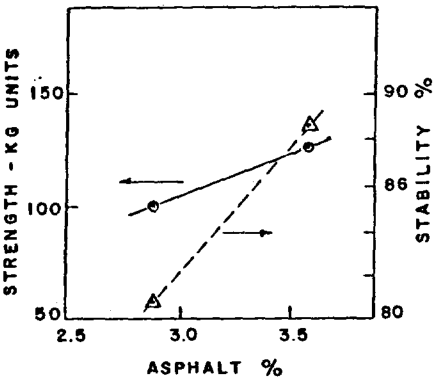


Fig. 2.

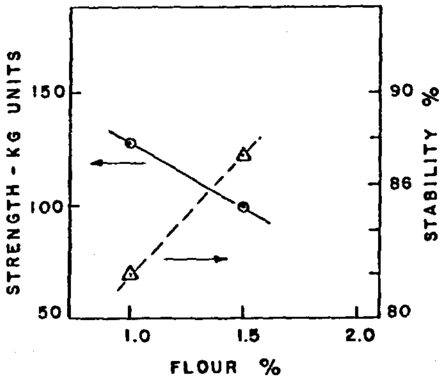


Fig. 3.

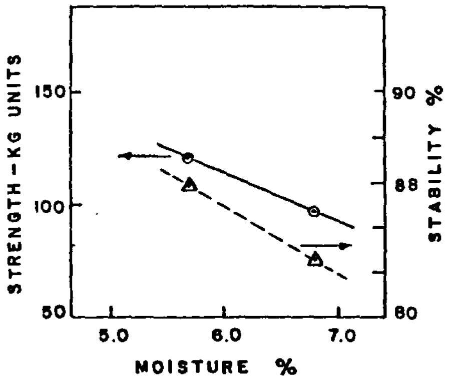


Fig. 4.

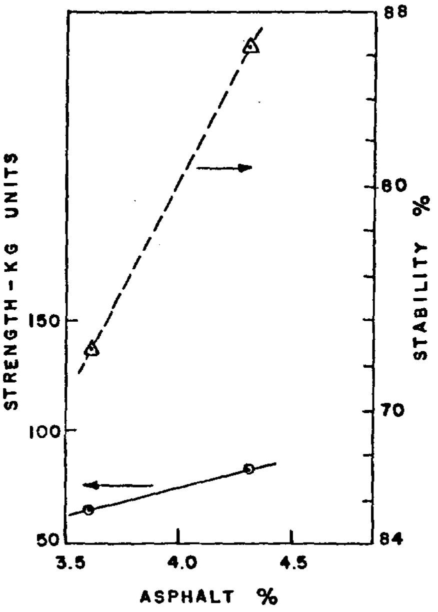


Fig. 5.

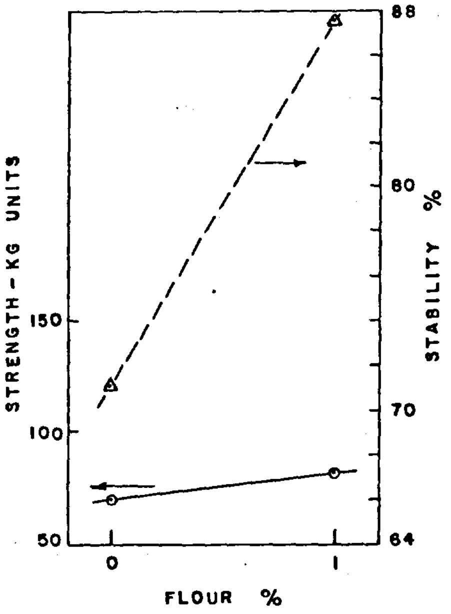


Fig. 6.

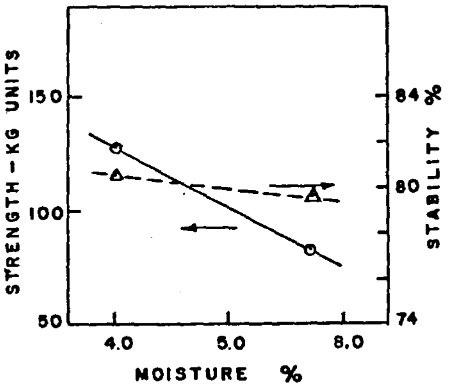


Fig. 7.

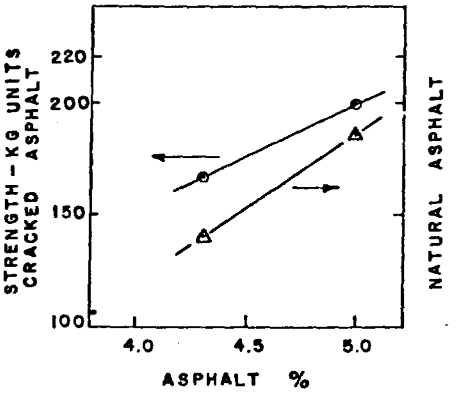
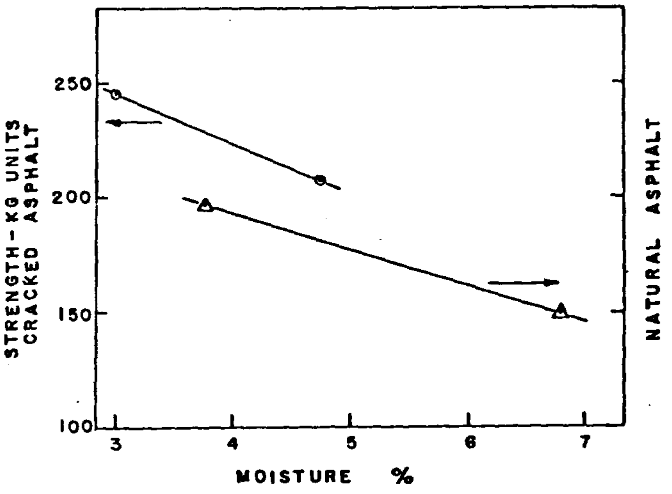


Fig. 8.



Figures 4, 5 and 6 show the variations in physical properties of the briquets in function of the percentages of asphalt, flour and moisture.

The third range of conditions extended to even higher asphalt contents: 4.3 - 5.0% and therefore no flour binder at all and a range of moisture content of 3.0 - 7.5%. In this case a comparison was made between natural straight run distillation asphalt and cracking asphalt.

Figures 7 and 8 show the variations in the compressive strength of the briquets in function of the percentage and nature of the asphalt and the moisture content.

The most important results may be briefly summarized as follows by simplifying as much as possible the conclusions:

(a) Effect of the asphalt binder: For every 0.1% increase in asphalt content, the compressive strength of the briquets increased by 3 to 4% and the tumbler stability increased by 1 to 2%. With the cracked asphalt, the compressive strength (after one hour of curing) was about 16% higher and the stability was about 7% lower than with the natural asphalt, but the difference in strength gradually disappeared with longer curing times and the difference in stability was apparent only when asphalt and moisture contents were both high or low.

(b) Effect of the flour binder: For every 0.1% increase in flour content, the tumbler stability of the briquets increased by about 1½% but their compressive strength decreased by 4% when the asphalt content was low (2.9 to 3.6%). The effect of the flour binder on the compressive strength at higher asphalt contents was not clearly established by the tests.

(c) Effect of the moisture content: For every 0.1% increase in moisture content, the compressive strength of the briquets decreased by ½ to 2½% but the effect on the tumbler stability could not be clearly determined.

CONCLUSION

As shown in the preceding example, which is considered in more detail in the Appendix, the application of a factorial design of experiments and of various statistical techniques permits the investigator to determine more precisely the respective influences of briquetting factors from data collected in a greatly reduced number of tests (in this case one-fourth the number of tests required by the classical methods of investigation). In addition, data analysis and comparisons can be made which would be impossible without the use of statistical methods such as regression and correlation analysis and tests of significance.

From one briquetting plant to another, conditions vary so much that it is most important that a separate investigation of the influence of the various factors of operation be conducted in each case, for conclusions reached in one study could not be directly applied to another plant without new tests.

It is hoped that the preparation of research programs by briquetting engineers may be facilitated by the descriptions given herein of applications of statistical methods to the investigation of the briquetting process.

ACKNOWLEDGMENTS

The investigations on briquetting mentioned here have been conducted in collaboration with Mr. C.F.J. Rozenhart and Miss J.L.M. Picard of the Fuels Division, and their contributions to this study are hereby gratefully acknowledged. The contributions to this paper of Mr. E. Swartzman of the Fuels Division are also greatly appreciated.

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APPENDIX

STATISTICAL ANALYSIS OF BRIQUETTING TESTS

The following data correspond to the example described in Section II of this paper. The tests were conducted on a factorial plan at two levels. For example, a series of tests was designed with three factors, asphalt X, flour Y and moisture Z, each being maintained as closely as possible at two levels:

X = 4.3% and x = 3.6% content of asphalt binder,
Y = 1% and y = 0% content of flour binder,
and Z = 5.7% and z = 4.0% average moisture content.

Compressive Strength

Table 1 shows the results of compressive strength tests on briquets obtained with the eight following combinations of the three factors under consideration: XYZ, XYz, XyZ, Xyz, xYZ, xYz, xyZ, xyz.

TABLE 1

Factors			Average Compressive Strength of Briquets (10 replicates for each test) (in Komarek-Greaves units)	Sums
Asphalt %	Flour %	Moisture %		
X	Y	Z	122.0	477.8
		z	123.0	
	y	Z	79.3	
		z	153.5	
x	Y	Z	65.5	378.0
		z	121.0	
	y	Z	68.5	
		z	123.0	
			Sum	855.8
			Average	107.0

The partial sums corresponding to the first factor (asphalt content X) are also shown in Table 1, and similar results for factors Y and Z are given in Tables 2 and 3.

TABLE 2

Factor	Average Strength	Sums
Y	122.0 123.0 65.5 121.0	431.5
y	79.3 153.5 68.5 123.0	424.3

TABLE 3

Factor	Average Strength	Sums
Z	122.0 79.3 65.5 68.5	335.3
z	123.0 153.5 121.0 123.0	520.5

Very often, the influence of one factor is not the same at all levels of another factor or several other factors. These factors are then said to be related; they "interact". Tables 4, 5 and 6 show the interactions of the first order between two of the three factors, by giving the partial sums corresponding to the elimination of the third factor for each combination of factors, such as XY, Xy, xY, etc. For instance, in Table 6, at a high moisture level (Z), the compressive strength shows an increase when the flour content is raised from (y) to (Y), whereas at a low moisture level (z), it shows a decrease with higher flour contents.

TABLE 4

Factors	X	x
Y	245.0	186.5
y	232.6	191.5

TABLE 5

Factors	X	x
Z	201.3	134.0
z	276.5	244.0

TABLE 6

Factors	Y	y
Z	187.5	147.8
z	244.0	276.5

The statistical analysis of variance of the experimental results is summarized in Table 7.

TABLE 7

Analysis of Variance of the Compressive Strength

Nature of the component of variance		Degrees of freedom of the component variance	Value of the component variance estimate
Effect, order of variance	Source, factor or interaction		
Single factor	X	2 - 1 = 1	1245
	Y	1	(7)
	Z	1	4287
Interaction of the first order	XY	1 x 1 = 1	(37)
	XZ	1	152
	YZ	1	652
Interaction of the second order	XYZ	1 x 1 x 1 = 1	664
Residual or error variance	Replication, sampling, testing, etc.	79 - 7 = 72	6.3
	Total	8 x 10 - 1 = 79	

All the component variance estimates have one degree of freedom and their significance can be tested against the residual variance with 72 degrees of freedom by comparing the respective variance ratios with the F values of the F test of Fisher-Snedecor:

at the 5% level of probability, F = 3.99,

and, at the 1% level of probability, F = 7.04.

All are found to be significant at the 1% level of probability except those written between brackets, namely the influence of the flour content and the interaction between asphalt and flour, which therefore are not statistically significant. The conclusions based on the study of this analysis of variance are given in Section II of this report (pages 18-22) and are illustrated in Figures 4, 5 and 6.

Abrasion Stability

The briquets obtained in this investigation were also tested on an A.S.T.M. Tumbler as described in Section II and the results are summarized in Table 8, which is similar in form to Table 1.

TABLE 8

Factors			Abradability Index % (average of 2 replicates)	Sums
Asphalt %	Flour %	Moisture %		
X	Y	Z	9.6	51.7
		z	11.0	
	y	Z	22.9	
		z	8.2	
x	Y	Z	13.9	106.1
		z	11.6	
	y	Z	34.4	
		z	46.2	

Partial sums for the influence of flour and moisture content are given in Table 9, similar to Tables 2 and 3.

TABLE 9

Main factor	Level of factor	Abradability Index sums
Y	Y	46.1
	y	111.7
Z	Z	80.8
	z	77.0

The interactions are illustrated in Tables 10, 11 and 12, similar to Tables 4, 5 and 6.

TABLE 10

Factors	X	x
Y	20.6	25.5
y	31.1	80.6

TABLE 11

Factors	X	x
Z	32.5	46.3
z	19.2	57.8

TABLE 12

Factors	Y	y
Z	23.5	57.3
z	22.6	54.4

The analysis of variance of the abradability index results is summarized in Table 13, similar to Table 7.

TABLE 13

Analysis of Variance of the Abradability Index

Variance component		Degrees of freedom	Estimate of the component variance
Effect	Source		
Single factor	X	2 - 1 = 1	369.9
	Y	1	537.9
	Z	1	(1.8)
Interaction of the first order	XY	1 x 1 = 1	248.7
	XZ	1	65.0
	YZ	1	(0.5)
Interaction of the second order	XYZ	1 x 1 x 1 = 1	114.0
Residual variance	Other factors	15 - 7 = 8	4.97
	Total	8 x 2 - 1 = 15	

The significance of the component variances can be tested against the residual variance by comparing the respective variance ratios with the F values of the F test:

at the 5% level of probability, F = 5.32,

and at the 1% level of probability, F = 11.26.

All are significant at the 1% level except those written between brackets (influence of moisture, and interaction between moisture and flour). The conclusions of this statistical analysis are given in Section II and are illustrated in Figures 4, 5 and 6.

Correlation, Regression and Index Formulae

On the basis of the test results obtained in a factorial plan of experiments, such as has been described above, it is often useful to calculate the coefficients of regression formulae giving an approximate value of the Komarek-Greaves Compressive Strength or the abrasion stability in function of the percentage content of asphalt X, flour Y or moisture Z, this function being assumed to be linear.

For instance, for tests within the ranges of:

asphalt	2.9 to 3.6%,
flour	1 to 1.5%, and
moisture	5.3 to 8%,

the following simple linear regression formula was computed for the compressive strength P:

$$P = 37.7X - 53.2Y - 24.1Z + 211.0$$

The correlation coefficient of this regression was good: 0.95 and the standard error was relatively low: 9 K-G units. For example, for

$$X = 3.6\%, \quad Y = 1.0\% \quad \text{and} \quad Z = 5.8\%$$

the formula gives P = 154 K-G. The observed value was 156.5. The calculated estimate will have a standard deviation of 9 K-G units. Similarly, an index formula can be calculated showing the influence of changes in the various factors in percentages: the formula corresponding to the case mentioned in the preceding paragraph was as follows:

$$I_P = 0.99I_X - 0.55I_Y - 1.09I_Z + 164$$

For example, when the asphalt content is increased by 10%, say from 3.6 to 4.0%, the compressive strength, P, increases by 9.9%, that is from 154 to 169 K-G units if Y = 1% and Z = 5.8%. Similarly, an increase of flour content from 1 to 1.1% decreases the compressive strength by 5.5% from 154 to 146 K-G units.

A regression formula such as was described above can be very useful, for instance in comparisons of various sets of combinations of factors. For example, a comparison was made between the use of a so-called emulsifier and a pneumatic cyclone nozzle atomizer.(3)

It was materially impossible to reproduce exactly the same conditions for the two types of equipment. The use of the regression formula given above was justified by the high value of the correlation coefficient. Therefore, tests with the emulsifier were conducted within the range of validity of this formula (contents of asphalt: 2.9 - 3.6%, flour: 1.0 - 1.5%, moisture: 5.3 - 8%) and the compressive strength values observed when using the emulsifier were compared with the values calculated for the atomizer by substituting in the regression formula the corresponding values of the factors X, Y and Z observed in each test, thereby obtaining Table 14.

TABLE 14

Test No.	Komarek-Greaves Compressive Strength (average of 10 replicates) in K-G units		
	With emulsifier (observed data)	With atomizer (values calculated from the regression formula)	Difference d
1	120	132	12
2	98	101	3
3	106	113	7
4	80	124	44
5	117	149	32
6	96	93	-3
7	130	127	-3
8	94	103	9
Sum	841	942	101
Average	105.13	117.75	$\bar{d}$ = 12.63

The differences d can be regarded as normally distributed, and, as the number of tests n is small (n = 8), the influence of the atomizer may be assessed by the t-test of Student-Fisher:

$$t = \frac{\bar{d} - 0}{s_{\bar{d}}} = \frac{\bar{d}}{\sqrt{\Sigma(d - \bar{d})^2}} \sqrt{n(n - 1)} = 2.12$$

with n - 1 = 7 degrees of freedom. The Student-Fisher t-test tables show that there is a probability of nearly 95% that the atomizer is significantly better than the emulsifier under the conditions considered here.

It should be noted that if the two averages (with emulsifier: 105.13 and with atomizer: 117.75) had been directly compared, as they should be if it could not be assumed that the same conditions prevailed with both types of equipment, then the additional information provided by the consideration of the differences d would not be used. In that case, as n is small, the variance $s_1^2 = 267.28$ of the data obtained with emulsifier must be compared by a test such as the F-test of Fisher-Snedecor with the variance $s_2^2 = 348.28$ of the data calculated for the atomizer. The F ratio is 1.3. With 7 degrees of freedom for each variance, there is no significant difference between them even at the 10% level of probability. Therefore the two averages can be compared by a t-test of Student-Fisher, using as an estimate of the common variance:

$$s^2 = \frac{(n - 1)\, s_1^2 + (n - 1)\, s_2^2}{(n - 1) + (n - 1)} = \tfrac{1}{2}(s_1^2 + s_2^2) = 307.79$$

Hence:

$$t = \frac{\bar{d}}{s}\sqrt{\frac{n}{2}} = 1.44 \quad \text{with } 2(n - 1) = 14 \text{ degrees of freedom}$$

and this analysis would fail to show a significant difference between the atomizer and the emulsifier even at the 17% level of probability.

If the two variances s_1^2 and s_2^2 had been found significantly different by the F-test, the averages could still have been compared by other tests such as the Behrens-Fisher-Sukhatme test, but not by the t-test. In the case where the means are actually not significantly different but the variances are different, the t-test might lead to a rejection of the Null hypothesis of identical means.

In conclusion, it should be emphasized that before applying any statistical technique which requires distributions to have certain characteristics, such as normality, constant variance, large number of observations, additivity of effects, etc., it must always be first ascertained that these conditions are fulfilled. Therefore the types and characteristics of the distributions of the data must be studied first, and transformations of the data and special designs of the experiments should be considered, if necessary, to permit the legitimate application of the most effective statistical methods.

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