Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

CANMET
Canada Centre for Mineral and Energy Technology
Centre canadien de la technologie des minéraux et de l'énergie

INVESTIGATION OF THE USE OF SASKATCHEWAN LIGNITE CHAR IN PITCH-BOUND FORMED COKE BRIQUETTES

M. SARACOĞULLARI, M.J. MALETTE AND W.R. LEEDER

FEBRUARY 1979

ENERGY RESEARCH PROGRAM
ENERGY RESEARCH LABORATORIES
REPORT ERP/ERL 79-4 (IR)

1. INTRODUCTION

Formed coke is the term used to describe carbonized or partially carbonized coal briquettes which have been prepared by a process including a mechanical shaping stage and which have been heated beyond the decomposition temperature of coal.

Many formed-coke processes have been developed to various stages although about eight of these may be considered to be close to commercial exploitation (1). The formed-coke processes being developed can be classified into three main categories by the type of binder used in briquetting (2): coking coal binder processes, pitch binder processes and binderless processes.

This report presents the results of a research study to investigate the possibility of producing mechanically a formed-coke from pitch bound briquettes made of Saskatchewan lignite char. Tests were performed in the Laboratory to determine the effects of process variables char size, pitch contents, oxidation time, temperature and air flow during oxidation, on the mechanical strength of green and oxidized briquettes. The mechanical strength of briquettes was determined using modified tumbling and crushing strength tests.

One batch of briquettes was made in the pilot-scale, using the best conditions obtained in laboratory study, to compare the strength of such formed coke produced from oxidized pitch-bound char briquettes with conventional coke.

2. EXPERIMENTAL

2.1 Materials:

The char used in briquettes were a commercial sample of Saskatchewan lignite char that had a volatile matter content of 14 percent. This value was thought to be too high to obtain mechanically strong briquettes, according to the results of earlier studies (3), and it is decided to apply a secondary carbonization to the char and to reduce the volatile matter to a target level of 3 percent.

The char was recharred by carbonizing it in an electrically heated rotary kiln in Energy Research Laboratories of CANMET. This kiln is an open-end rotating retort, 6 ft. long 16 in. ID., which is electrically heated from the outside to give a hot zone 4 ft. long. The thermocouple for controlling the temperature in the furnace is between the retort and furnace wall. A loosely fitting plug, placed in the cold end of retort, has a 2.5 in diameter hole in the middle of it through which the volatiles escape and are burned. An exhaust hood was located near the retort to provide draught to remove the combustion products.

The original char volatile matter content was determined thermogravimetrically, Figure 1. Charring tests were then done to check char volatile content change with the furnace temperature and carbonization time. The lowest volatile content, 4.5 percent, was achieved at a furnace temperature of 1700°F with a carbonization time of one and half hours. The char for the study was produced at these conditions. The proximate analysis of samples charred for different times at 1700°F, raw char and the final char sample used in the research studies are given Tables 1 and 2.

A coal-derived pitch was used as the binder. This pitch has an ASTM ring and ball apparatus softening point of 58°C (4). The choice of this binder was to have a low degree of aromatization that is chemically reactive to oxygen. Such a material has been shown to be useful as a binding material for this type of formed coke process (5). The coal-derived, low softening temperature pitch was used in this research study was believed to meet these requirements.

2.2 <u>Briquetting:</u>

Briquettes were made by the following procedure. At least 30 lbs. of char were placed in a mixer heated from steam jacket. The mixer was started and about 20 percent water added to suppress the dust and aid binder distribution. Steam was passed through a steam jacket around the mixing chamber to heat the char water mixture to 50-70°C. The required quantity of melted pitch was slowly poured into the blending mixture and mixing was continued for 15 minutes. The warm mixture was then poured onto the floor and allowed to cool. When the temperature was between 140°F and 150°F, the mixture was briquetted by pouring it down a vertical chute into an operating Komarek-Greaves double roll press, from which 3.8 x 3.8 x 1.4 cm pillow-shaped briquettes were produced. The warm briquettes were allowed to cool to room temperature.

A preliminary study has been done to determine the best size distribution of char. Different proportions of fine and course chars were briquetted using manual briquetting press. In these tests 15 percent binder and about 20 percent water was used. A pressure of 4817 lbs/in^2 was applied.

2.3 Oxidation:

Oxidation was done by heating at least 10 briquettes for 0.5-2 hours at 220°C - 280°C in a muffle furnace. Air preheated to the temperature of the furnace was allowed to flow over the briquettes. To check the temperature and detect any combustion, a thermocouple was located above the briquettes. The rate of air flow in the muffle furnace was one of parameters which were investigated. After oxidation the briquettes were allowed to cool down to ambient temperature for measuring mechanical strengths.

2.4 Measurement of Mechanical Strength of Briquettes:

2.4.1 Crushing Strength:

Crushing strengths of green and oxidized briquettes, were determined using a Rainhart Crushing Machine which has two parallel plate closing at a rate of 0.05 in/min. At least 6 briquettes were crushed and the average value was reported as the crushing strength.

2.4.2 Tumble Test:

Laboratory scale studies were unable to produce enough briquettes to carry out a ASTM coke tumble test (6). To evaluate the mechanical tumble quality of the briquettes, a small laboratory modified tumbler was used. This test consisted of tumbling 5 briquettes at 80 rpm in a 4 in. diameter steel drum equipped with three equally-spaced 0.75 in. lifters. After 15 and 30 minutes tumbling, the tumbled materials were screened on 10 mesh and 0.5 in. sieves. The percentage by weight passing 0.5 in. sieve was reported as an abradability index for each 15 and 30 minutes tumbling.

Only one test was done at pilot scale level and the ASTM coke tumble test (6) was used. Twenty-two lbs. of oxidized briquettes was tumbled for 1400 revolutions at 24 ± 1 rpm in a 3 ft. diameter by 1.5 ft. long cylindrical drum equipped with two equispaced 2 in. lifters set at 90 degrees to the drum wall. Unfortunately the size of the briquettes were too small to be convenient for this test and consequently only the abradability index determined by the weight percent retained on 0.25 in. sieve was determinable.

3. RESULTS AND DISCUSSION

The effect of char size consist, amount of binder and oxidation conditions on crushing strength and tumbling results of green and oxidized briquettes was investigated. This section will discuss the results.

The influence of char size on briquette density and crushing strength was investigated by making briquettes manually in a laboratory press. As the fine char content is increased in the briquetting material, density and crushing strength of briquettes increased, Figure 2. Briquetting using the roll press used four different char size consists: 75 percent minus 6 mesh, 100 percent minus 6 mesh, 82 percent minus 20 mesh and 100 percent minus 12 mesh. The latter was suggested to be the best char size from the manual press tests. The size analyses of the chars appears in Figure 3. Binder content was varied from 7 percent to 15 percent. A summation of the results of the roll-press tests is seen in Table 3. The variations of crushing strength and abrasion index of green briquettes produced from 100 percent minus 12 mesh, with the amount of binder used, is given in Figure 4. As could be expected, increased pitch binder content in green briquettes increased the crushing strength and reduced abrasion ability.

The effect oxidation parameters such as oxidation time, oxidation temperature and air flow rate on crushing strength and tumbler test results, have been investigated on the briquettes produced by using 100 percent minus 12 mesh char and 10, 12.5 and 15 percent binder. Oxidation temperature is varied from 220°C to 280°C. Figure 5 and 6 show the influence of oxidation temperature and binder content of briquettes on the crushing strength and abrasion index respectively. Crushing strength increases significantly with oxidation temperature for all binder content. The abrasion index decreases slightly with oxidation temperature but the most significant decrease was obtained by increasing the amount of binder.

The data have been replotted in Figure 7 and 8 to show the influence of oxidation time and binder content on crushing strength and abrasion index of oxidized briquettes respectively. In this investigation, the binder content of briquettes was found to be the most important variable that effects briquette mechanical strengths.

Another parameter investigated in oxidation is air flow rate used during oxidation. It was varied from 0.48 cu ft/min to 1.0 cu ft/min.

Increased air flow rate increased the crushing strength, especially in the oxidized briquettes made with 15 percent binder. The effects of air flow rate on briquette crushing strength and abrasion have been plotted in Figures 9 and 10.

A relationship was found between crushing strength and abrasion index of oxidized briquettes in both 15 minutes and 30 minutes tumbling. According to data plotted in Figure 11 abrasion index of briquettes decreases as the crushing strength increases.

4. CONCLUSIONS

The percentage binder content and char size consist were found as major parameters that effect mechanical strength of green and oxidized briquettes.

In this study 15 percent binder gave the highest mechanically strength briquettes with the lignite char tested. This binder percentage may be reduced using higher rank coal char or by charging the char size consist briquette size, and/or briquetting pressure.

The best conditions for oxidation of pitch bound briquettes was found to be a temperature of 280°C, an oxidation time of 1.5 hours and an air flow rate of 1 cu ft/min.

One pilot-scale batch test, using the best conditions found in the laboratory study, has been done to prepare enough briquettes to compare their abrasion index with metallurgical coke, according to ASTM tumbler test method. The abrasion index, hardness factor, of the final product was found to be 34 percent. This value is very low as comparison with good metallurgical coke. However the briquettes were not carbonized and were too small to be suitable for the ASTM test. For a more accurate comparison it is necessary further investigations with the briquettes which have dimensions of about 2 in. by 3 in. and which have been carbonized.

ACKNOWLEDGEMENTS

The authors wish to thank the UNIDO and CIDA for support of Mr. Saracoğullari during his program.

REFERENCES

1. Fisher A.D. "Metalurgical coals for the future". 23rd Canadian Conference on Coal; 1971.
2. Leeder, W.R. and Whalley, B.J.P. "Formed coke activities with Canadian coals". CANMET Report ERP/ERL 75-152; Dec.; 1975.
3. Leeder, W.R., Malette, M.J. and Silver, M.M. "Formed-Coke program study". CANMET Report ERP/ERL 76-1979 (tr); Nov.; 1973.
4. ASTM Designation: D36-70 Annual Book of ASTM Standards; American Society for Testing and Materials, Philadelphia; Part II; 1973.
5. Zielinski, H. "Present methods of coke manufacuture". pp. 218; 1972.
6. ASTM Designation D294-64 Annual Book of ASTM Standards; American Society for Testing and Materials, Philadelphia; 1973.

TABLE 1 Proximate Analysis of Char Samples taken during carbonization at 1700°F in Rotary Kiln.

	After 60 min.	After 120 min	After 180 min	After 240 min	After 300 min	After 360 min	Product Bulk Sample
Moisture ...... %	0.11	0.30	0.23	0.44	0.09	0.57	0.27
Ash .... %	15.88	16.85	17.27	20.68	28.35	22.05	17.94
Volatile ...... %	6.83	4.34	3.69	4.14	4.21	5.04	4.42
Fixed C %	77.18	78.51	78.81	74.74	67.35	72.34	77.37

TABLE 2 Proximate Analysis of Raw Materials

		Raw Char	Carbonized Char
Moisture	%	10.83	1.50
Ash	%	12.50	16.39
Volatile	%	14.33	4.81
Fixed C	%	62.34	77.30

TABLE 3 Influences of char size consist and binder percent on the crushing strength of green briquettes.

BRIQUETTED MATERIALS		CRUSHING STRENGTH OF GREEN BRIQUETTES (lbs)
SIZE CONSIST OF CHAR	BINDER %	
-6 mesh 75%	7	nil
-6 mesh 75%	10	124
-6 mesh 100%	10	91
-6 mesh 100%	13	141
-20 mesh 82%	10	144
-20 mesh 82%	15	331
-20 mesh 82%	10*	61
-12 mesh 100%	10	160
-12 mesh 100%	12.5	213
-12 mesh 100%	15	310

* In this test a different pitch was used as the binder.

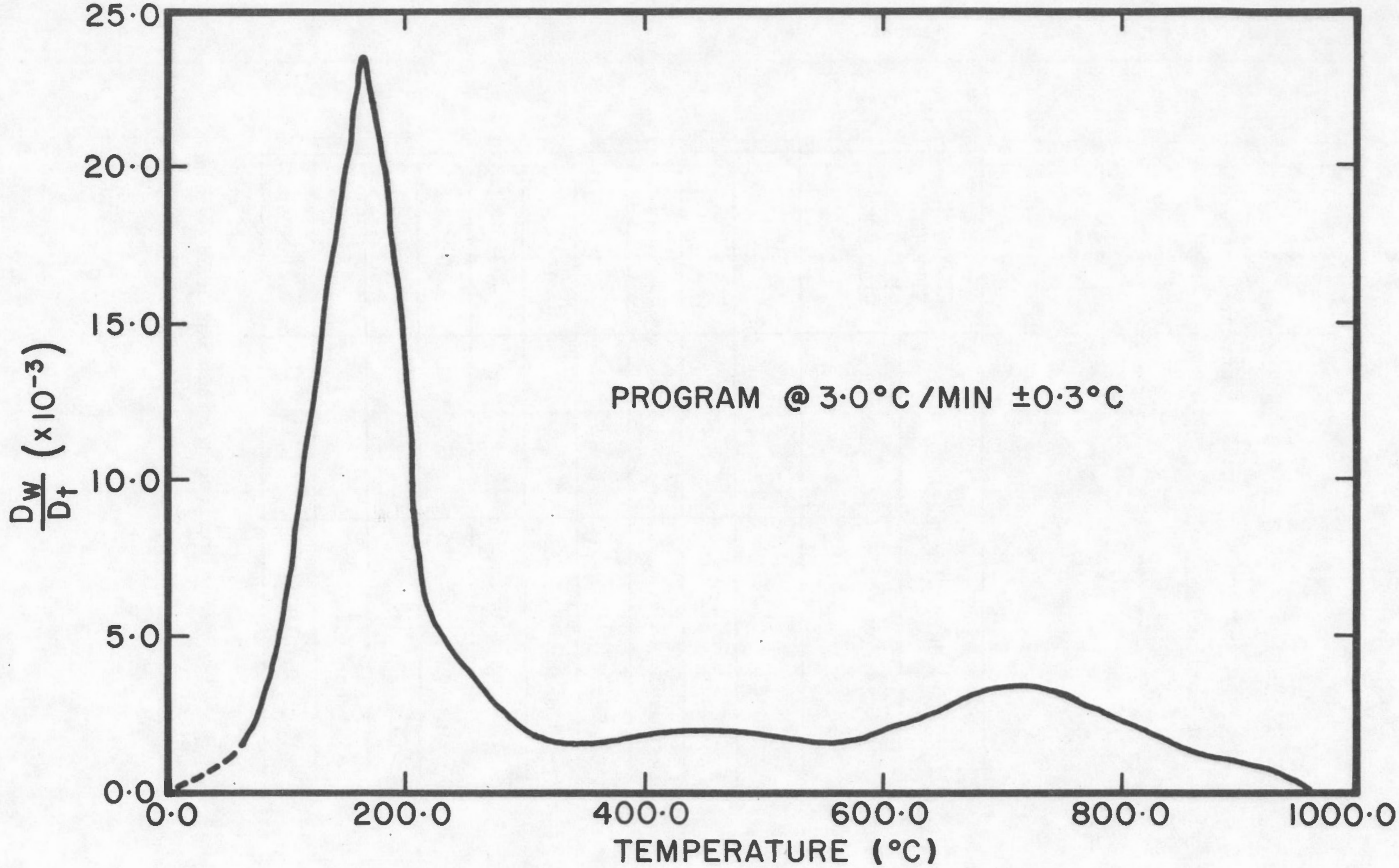

FIGURE 1 Thermo-gravimetric analysis of Saskatchewan Lignite char

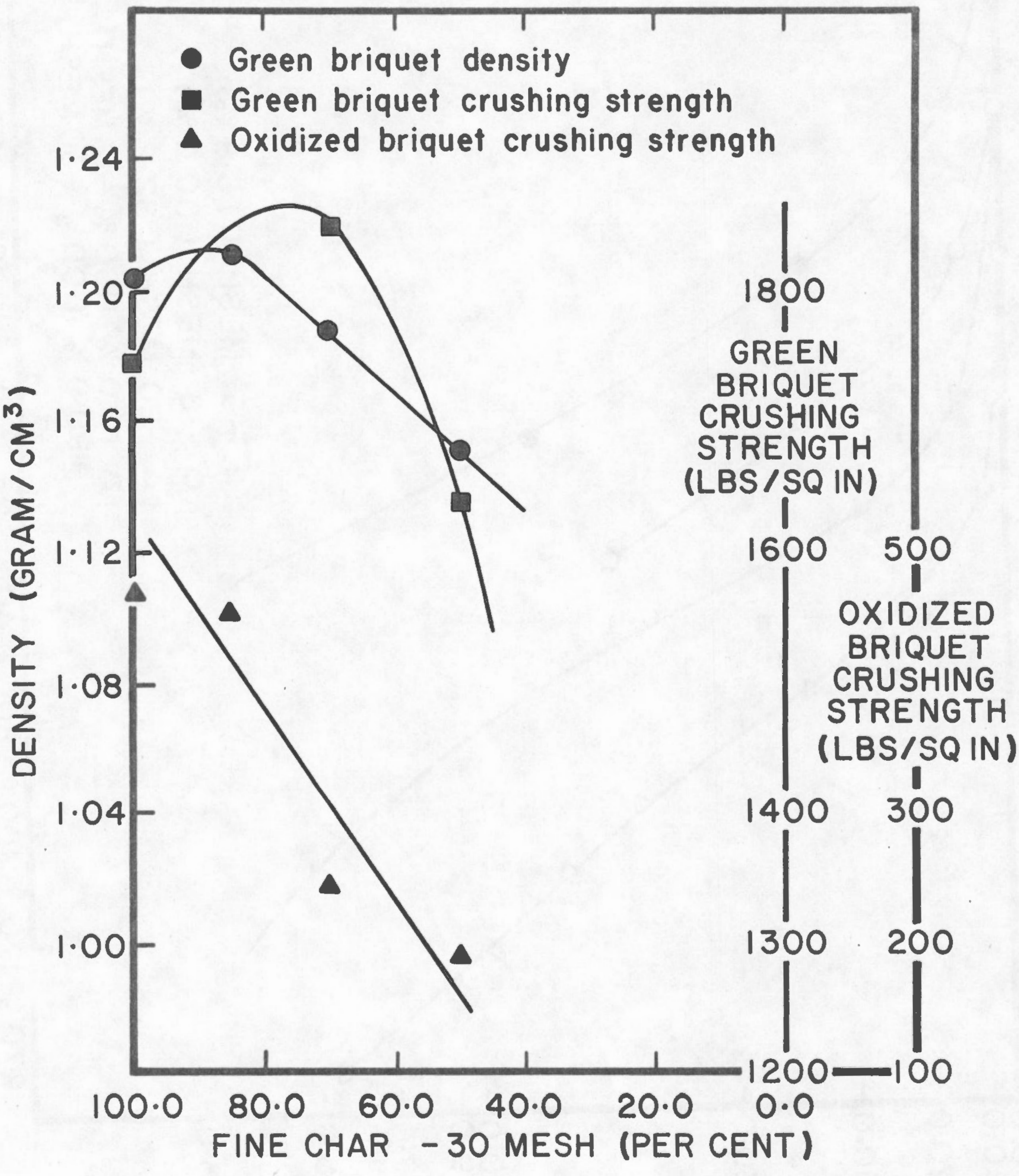

FIGURE 2 The effects of char size on the crushing strength and density of green and oxidized cylindrical briquettes produced manually in the laboratory.

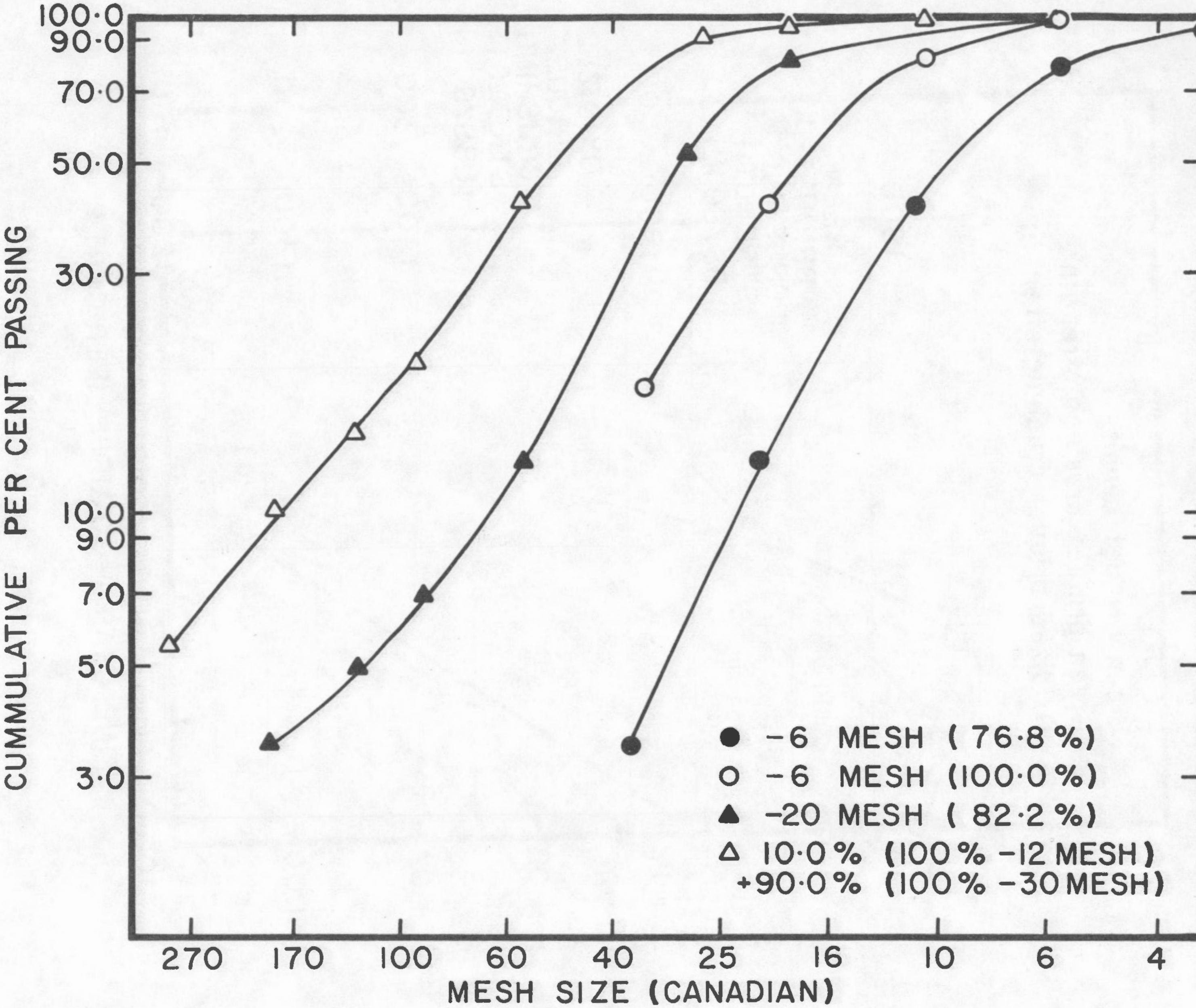

FIGURE 3 Screen analysis of material used in briquetting

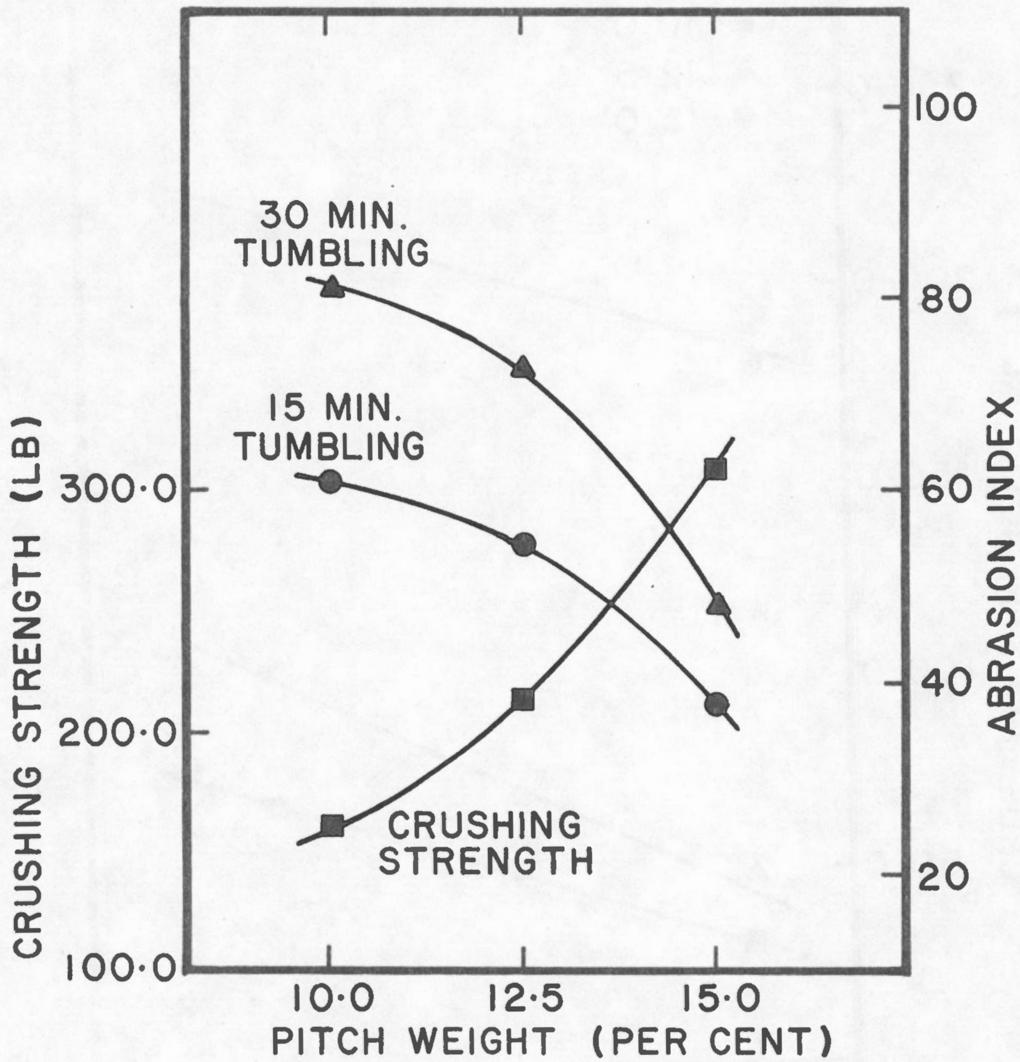

FIGURE 4 Effects of percentage pitch content on crushing strength and abrasion index of green briquettes produced in the roll press.

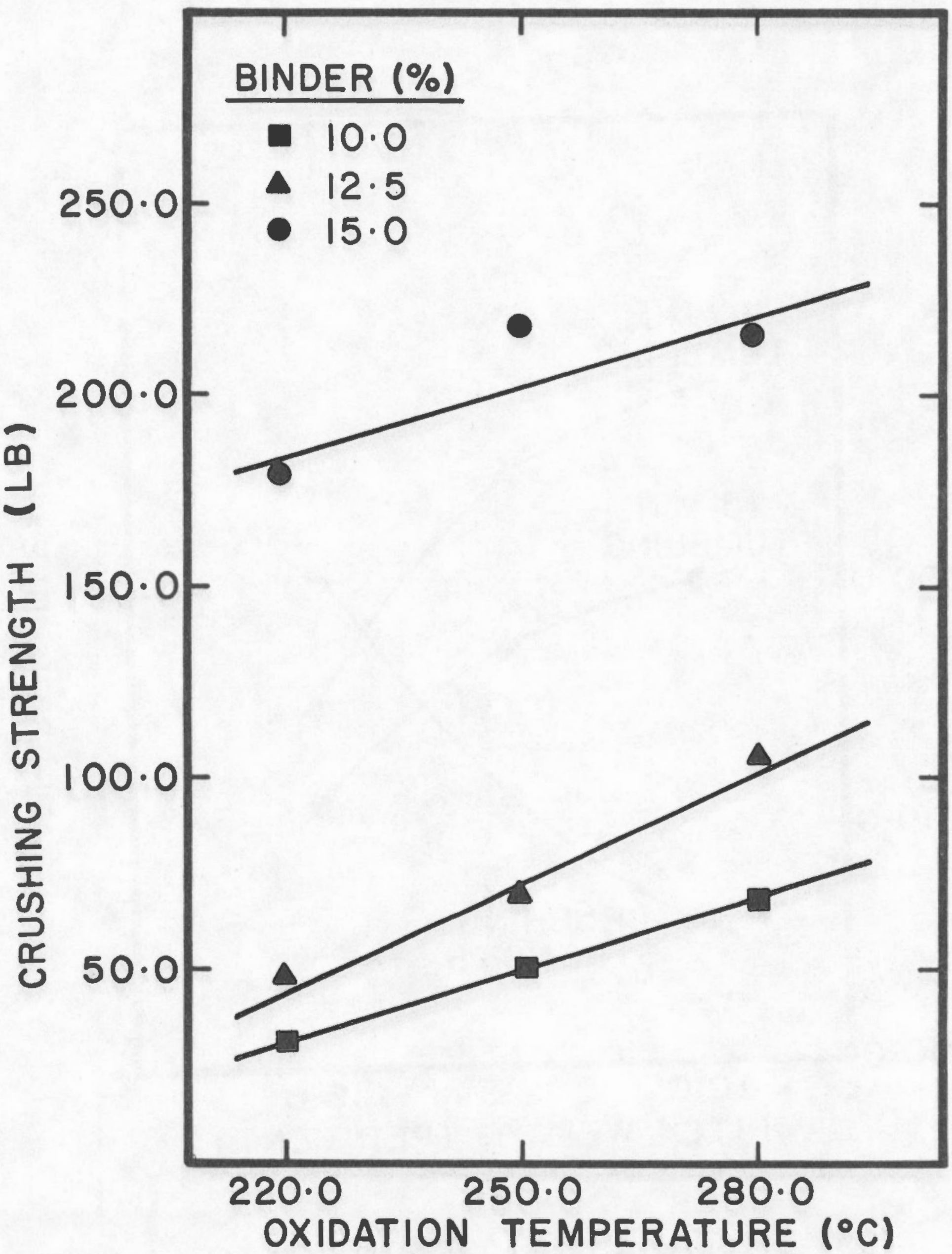

FIGURE 5 Effect of oxidation temperature on crushing strength of oxidized briquettes- (roll-press briquettes - oxidized for 2 hrs/ 1 cu. ft. air flow/220 - 280°C)

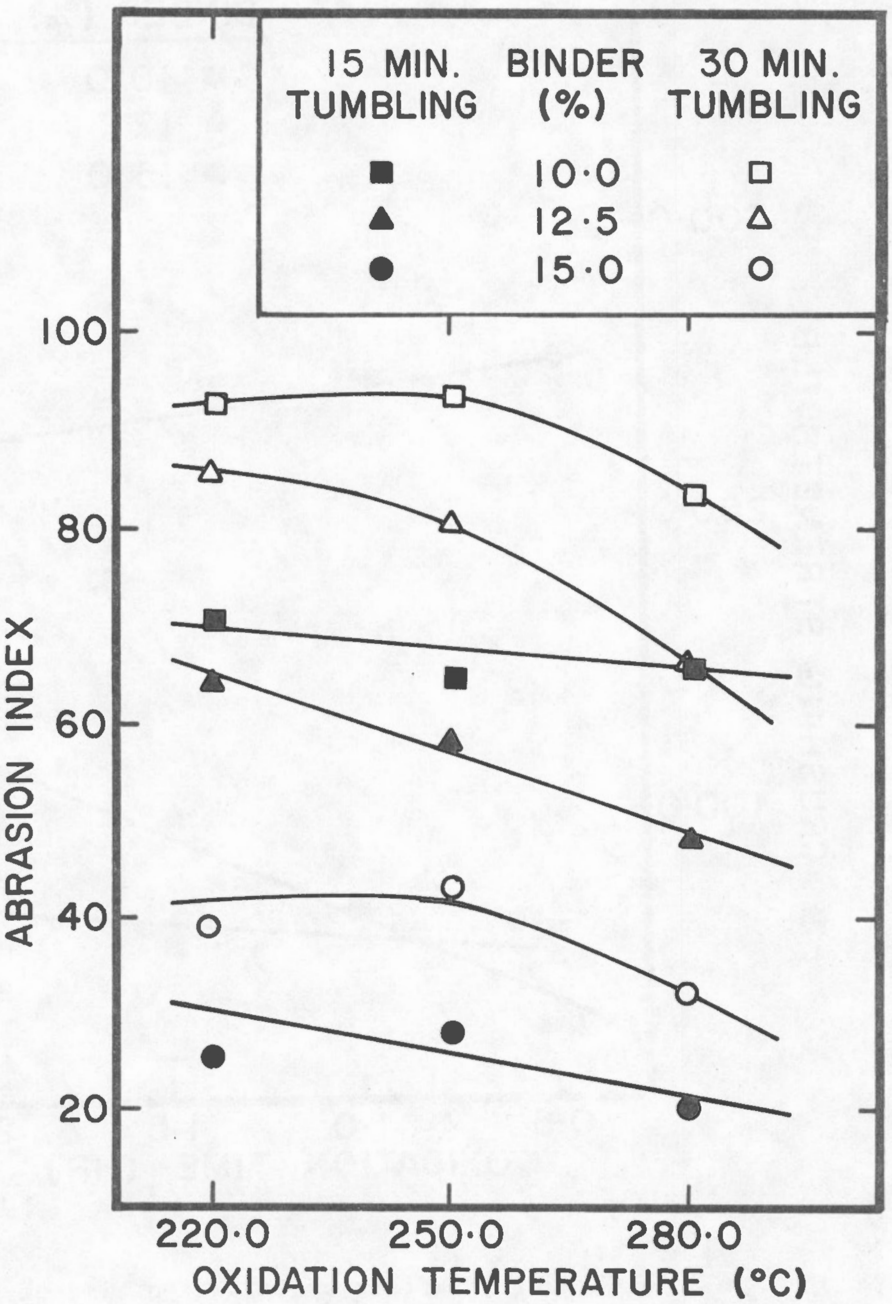

FIGURE 6 Effect of oxidation temperature on abrasion index of oxidized briquettes (roll-press briquettes - oxidized for 2 hrs/1 cu. ft. air flow/220 - 280^{o}C).

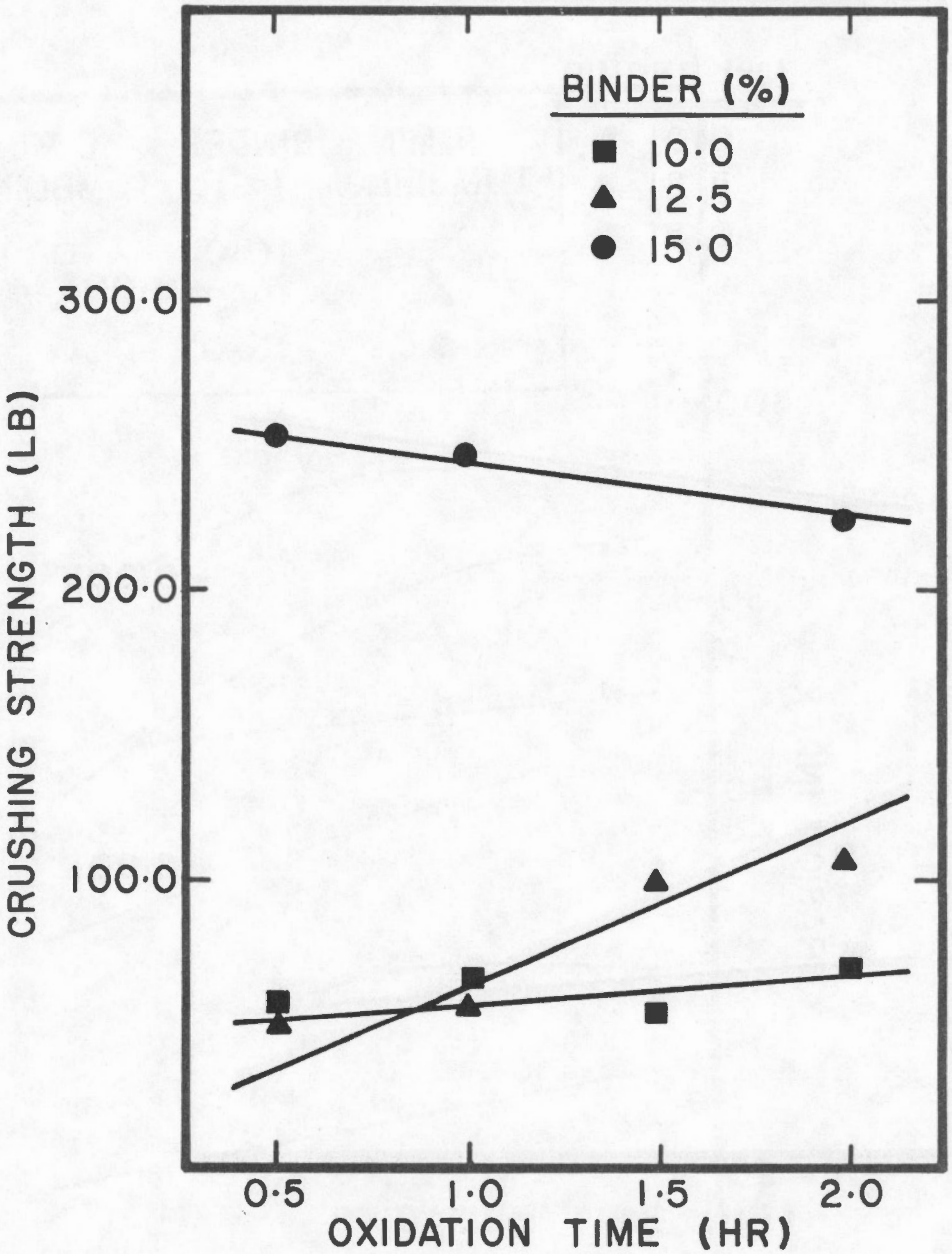

FIGURE 7 Effect of Oxidation time on crushing strength of oxidized briquettes (roll-press briquettes - oxidized at $280^{\circ}C$/1 cu. ft. air flow/0.5-2 hrs).

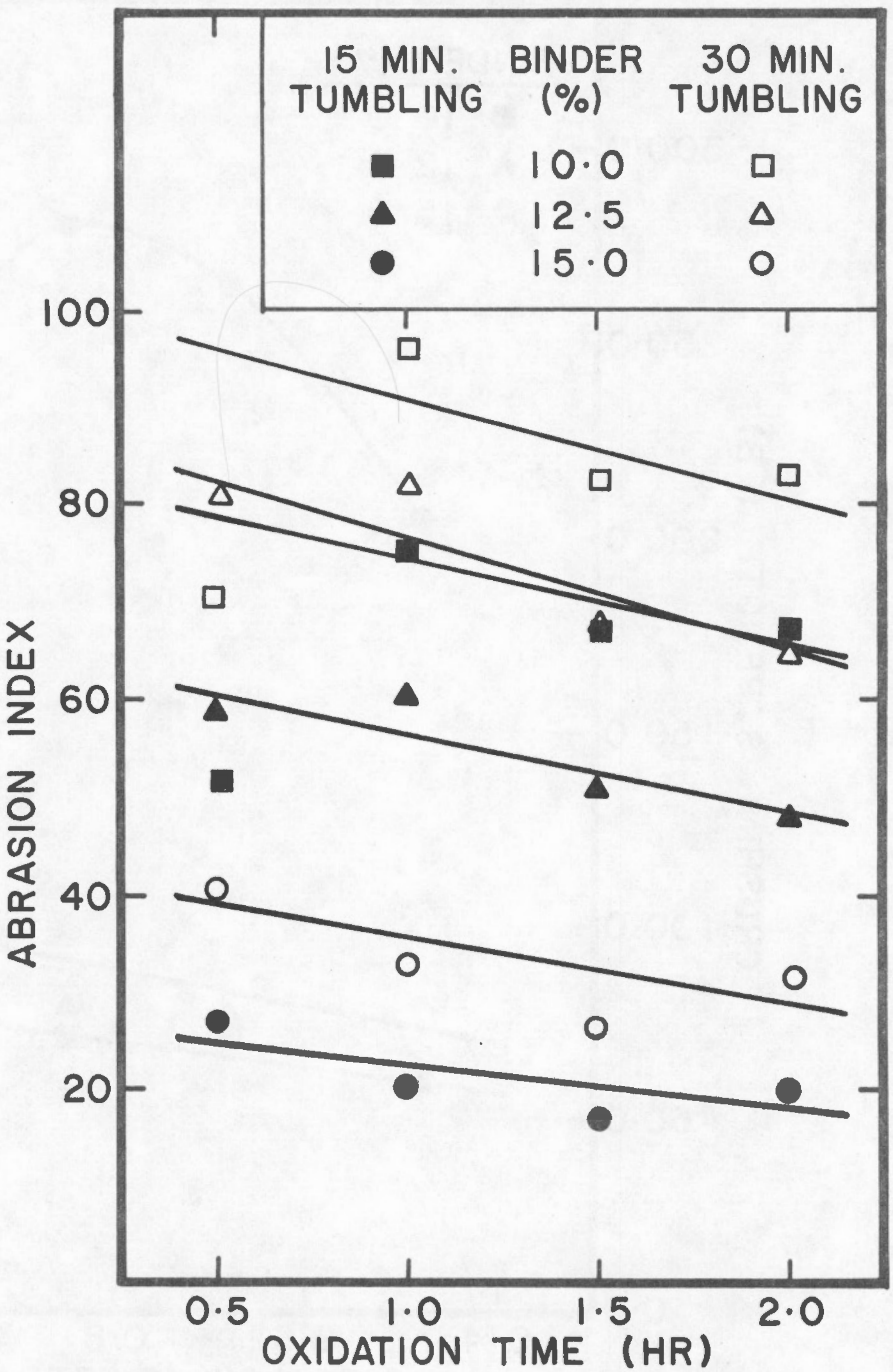

FIGURE 8 Effect of oxidation time on abrasion index of oxidized briquettes (roll-press briquettes - oxidized at 280°C/1 cu. ft. air flow/0.5-2 hrs.).

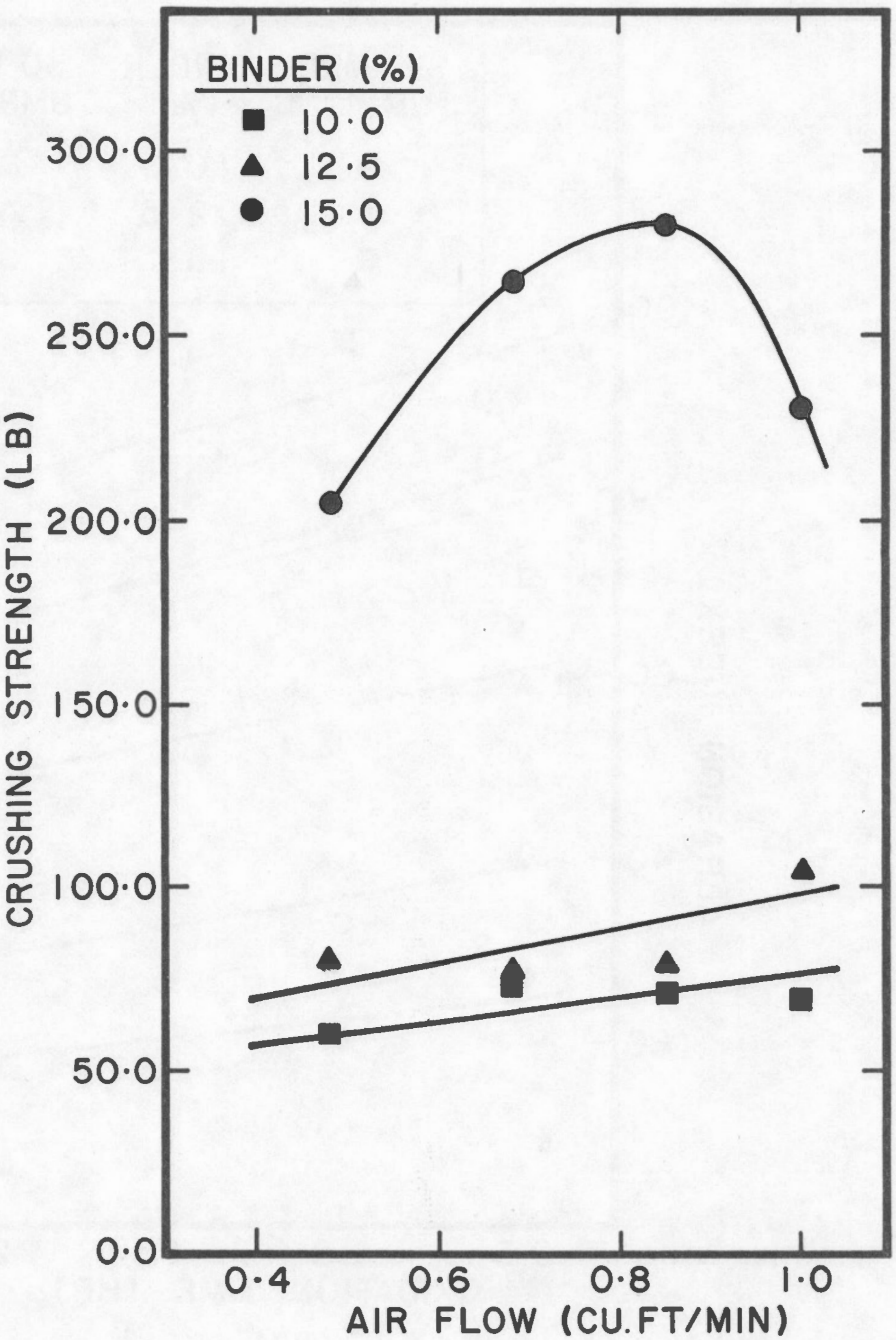

FIGURE 9 Effect of air flow rate on crushing strength of oxidized briquettes (roll-press briquettes - oxidized at 280°C/2 hrs./0.48 - 1.00 cu. ft. air flow).

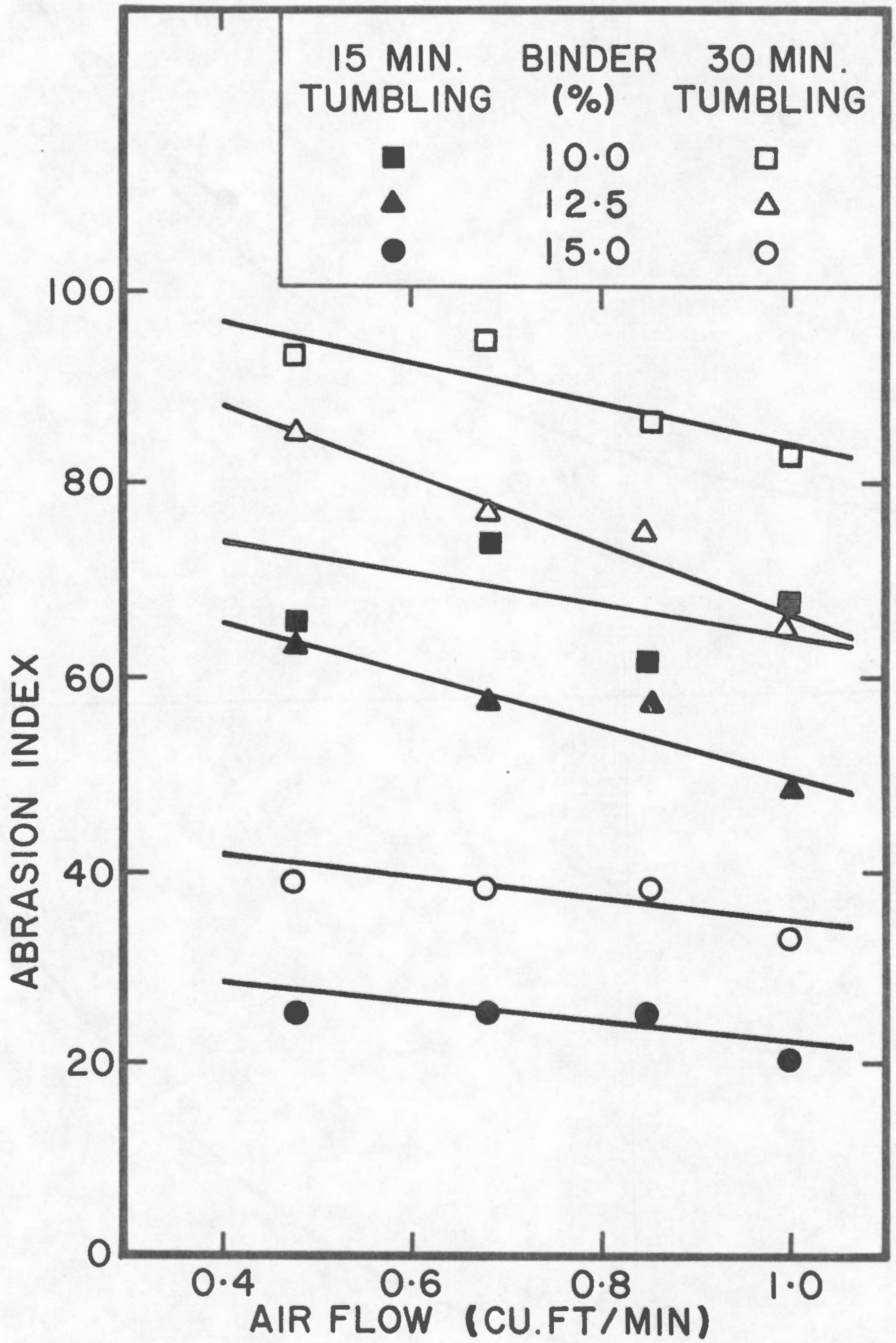

FIGURE 10 Effect of air flow rate on abrasion index of oxidized briquettes (roll-press briquettes oxidized at 280°C/2 hrs./0.48 - 1.00 cu. ft. air flow).

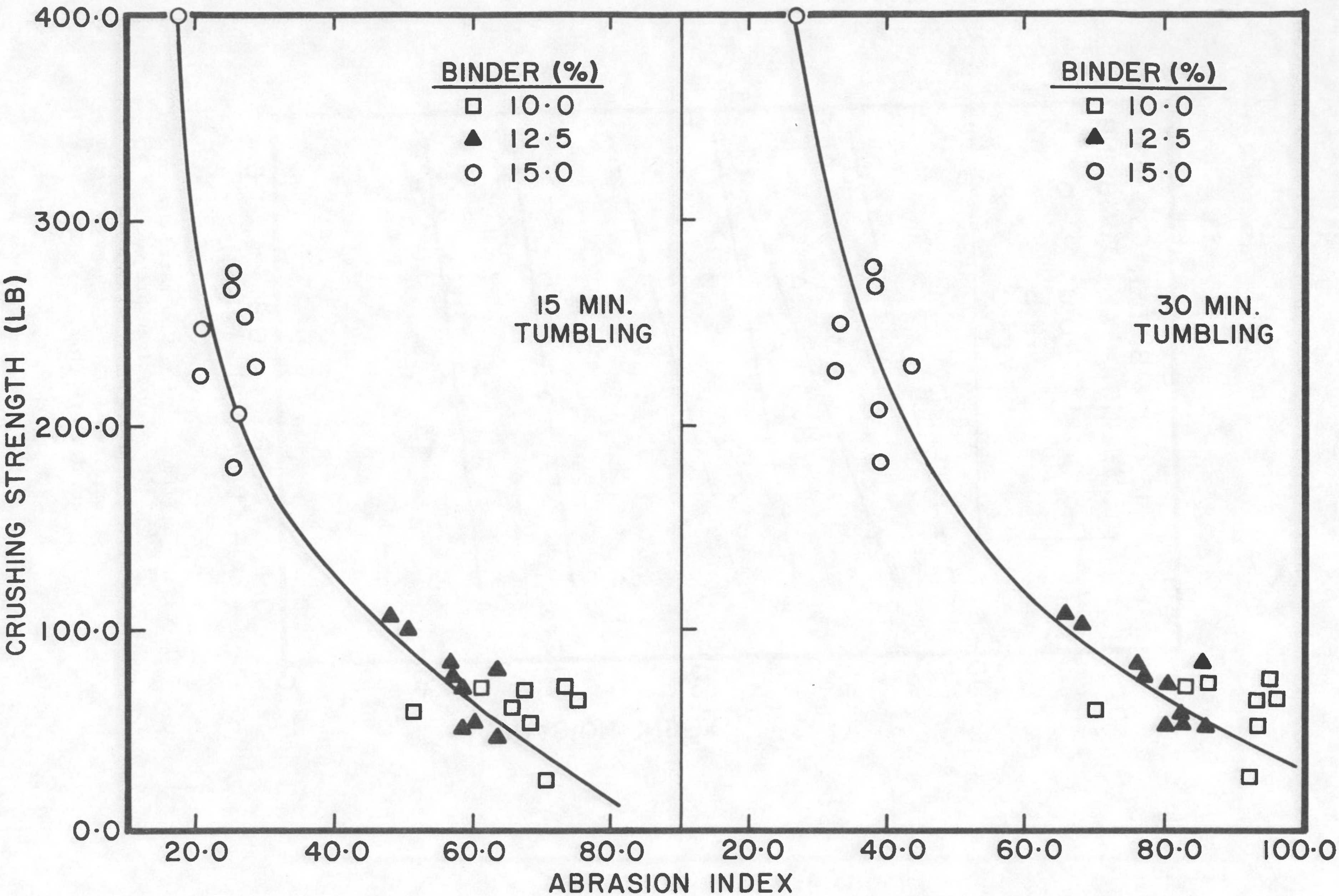

FIGURE 11 The relation between crushing strength and abrasion index of oxidized briquettes.