CANADA

DEPARTMENT OF ENERGY, MINES AND RESOURCES

OTTAWA

MINES BRANCH INVESTIGATION REPORT

IR 69-37

June, 1969 K1

ASSESSMENT OF A SHALE SAMPLE FROM TABER, ALBERTA, AS RAW MATERIAL FOR LIGHTWEIGHT AGGREGATE

by

H.S. Wilson & J.A. Soles

Mineral Processing Division

Note: This report relates essentially to the sample as received, the report and any correspondence connected therewith shall not be used in full or in part as publicity or advertising matter.

Mines Branch Investigation Report IR 69-37

ASSESSMENT OF A SHALE SAMPLE FROM TABER, ALBERTA, AS RAW MATERIAL FOR LIGHTWEIGHT AGGREGATE

by

H.S. Wilson* and J.A. Soles*

SUMMARY OF RESULTS

The sample was composed predominantly of a brown shale, and lesser amounts of blue shale. The blue fraction bloated more and through a wider temperature range than did the brown fraction.

Petrographic and X-ray analyses showed the blue shale contained a greater proportion of fine-grained, easily-fusible clay minerals and fewer coarse-grained refractory minerals than did the brown shale. This explains the better bloating characteristics of the former.

The shales should be fired either separately in lump form, or ground, mixed and fired in pelletized form. Satisfactory lightweight aggregate could probably be produced by either method, the latter being preferable.

---

* Research Scientists, Mineral Processing Division, Mines Branch, Department of Energy, Mines and Resources, Ottawa, Canada.

INTRODUCTION

In February, 1969, Taber Sand and Gravel Limited, Taber, Alberta submitted a 25-lb sample of shale for evaluation as a raw material for a lightweight aggregate. The sample was reported to have been taken from one of the company's pits in the vicinity of Taber.

The sample consisted predominantly of brown, silty shale in lumps up to about 4 inches. One lump of blue, finer-grained shale was included; it constituted about 10 per cent of the total sample.

The investigation consisted of stationary-kiln and rotary-kiln tests to observe the bloating characteristics of the shales, and petrographic examination and X-ray analysis to determine their mineralogy and textures.

STATIONARY-KILN TESTING

Preparation of Material

The sample was crushed to minus 1/2-inch size by means of a jaw crusher. Material for the tests was prepared in four different ways:

1. A representative portion was selected from the 1/4- to 1/2-inch fraction that had been obtained by hand screening.

2. Pieces of the two different shales in the sample were hand-picked from the 1/4- to 1/2-inch fraction.

3. The minus 1/4-inch material was further ground to minus 14 mesh, and a representative portion was brought to the plastic state by thoroughly mixing it with 25 per cent water. Spherical pellets 1/4 to 1/2 inch in diameter were formed by rolling pieces of the plastic shale between the palms of the hands.

4. Cylindrical pellets were formed by extruding the plastic shale from a hand-driven extrusion machine. The shale was put into the barrel of the machine in small pieces, the feed opening and the die openings were sealed and the material was de-aired by a vacuum pump. The piston in the machine was then moved forward to extrude the shale through the die openings. The streams, which were 3/8 inch in diameter, were cut into lengths of about 3/8 inch. The operation was repeated until a sufficient number of pellets had been formed.

All material thus prepared for testing were dried at 110°C (230°F).

Stationary-Kiln Firing

These tests were made in a gas-fired, up-draft kiln having a 4x8-inch hearth. About 15 grams of prepared material was used for each firing. The material was inserted into the preheated kiln, in a preheated shallow sagger, at temperatures of 1036°, 1063°, 1093°, 1120°, 1149°C, (1900°, 1950°, 2000°, 2050° and 2100°F). The retention time in the kiln was 8 minutes at each temperature.

After removal from the kiln, each product was examined for degree of bloating, and the bulk specific gravity was determined. For the specific gravity determinations, the weight was measured on a single-pan direct-reading balance and the volume was measured by the mercury-displacement method.

Results

The results of these tests are recorded in Table 1. The bulk specific gravities of the products are shown graphically in Figures 1 and 2. Figure 1 compares the brown and blue shales, in lump form, when fired together and when fired separately. Figure 2 compares the brown plus blue shale when fired in lump form, when fired in hand-pelletized form, and when fired in extruder-pelletized form.

Observations

The two shales comprising the sample exhibited different bloating characteristics. The brown shale bloated somewhat non-uniformly and to a lesser degree than did the blue shale, which bloated uniformly. The brown shale bloated appreciably between 1093° and 1149°C (2000° and 2100°F). The agglomerating temperatures of the two shales were about the same (1149°C or 2100°F).

Pelletizing the mixture of the two shales eliminated the non-uniformity of bloating. The method used to pelletize the mixture had little effect on the degree of bloating. The bulk specific gravities of the products in pellet form were about mid-way between those of the brown and blue shales fired separately in lump form.

TABLE 1

Stationary-Kiln Tests

Form	Unfired Bulk Sp Gr	Firing Temperature (°C)	Fired Bulk Sp Gr	Remarks
Lump: Brown plus blue	2.17	1036	1.70	Unbloated to slightly bloated
		1063	1.62	Unbloated and moderately bloated
		1090	1.23	Slightly bloated and very well bloated
		1120	0.87	Slightly bloated and very well bloated
		1149	0.70	Moderately bloated to over-bloated; severe sticking
Lump: Brown	2.15	1036	1.80	Unbloated to very slightly bloated
		1063	1.74	Unbloated to slightly bloated
		1093	1.45	Slightly to well bloated
		1120	1.09	Slightly to well bloated
		1149	1.02	Moderately bloated to over-bloated; moderate sticking
Lump: Blue	2.30	1036	1.48	Moderately bloated
		1063	1.08	Well bloated
		1093	0.76	Well bloated
		1120	0.47	Well bloated
		1149	0.31	Very well bloated; moderate sticking
Hand Pelletized	1.94	1036	1.63	Slightly bloated
		1063	1.36	Moderately bloated
		1093	1.11	Well bloated
		1120	0.83	Well bloated
		1149	0.63	Very well bloated; slight sticking
				Some pellets split at all temperatures
Extruder Pelletized	1.98	1036	1.66	Very slightly bloated
		1063	1.44	Slightly bloated
		1093	1.15	Well bloated
		1120	0.82	Well bloated
		1149	0.57	Very well bloated; moderate sticking

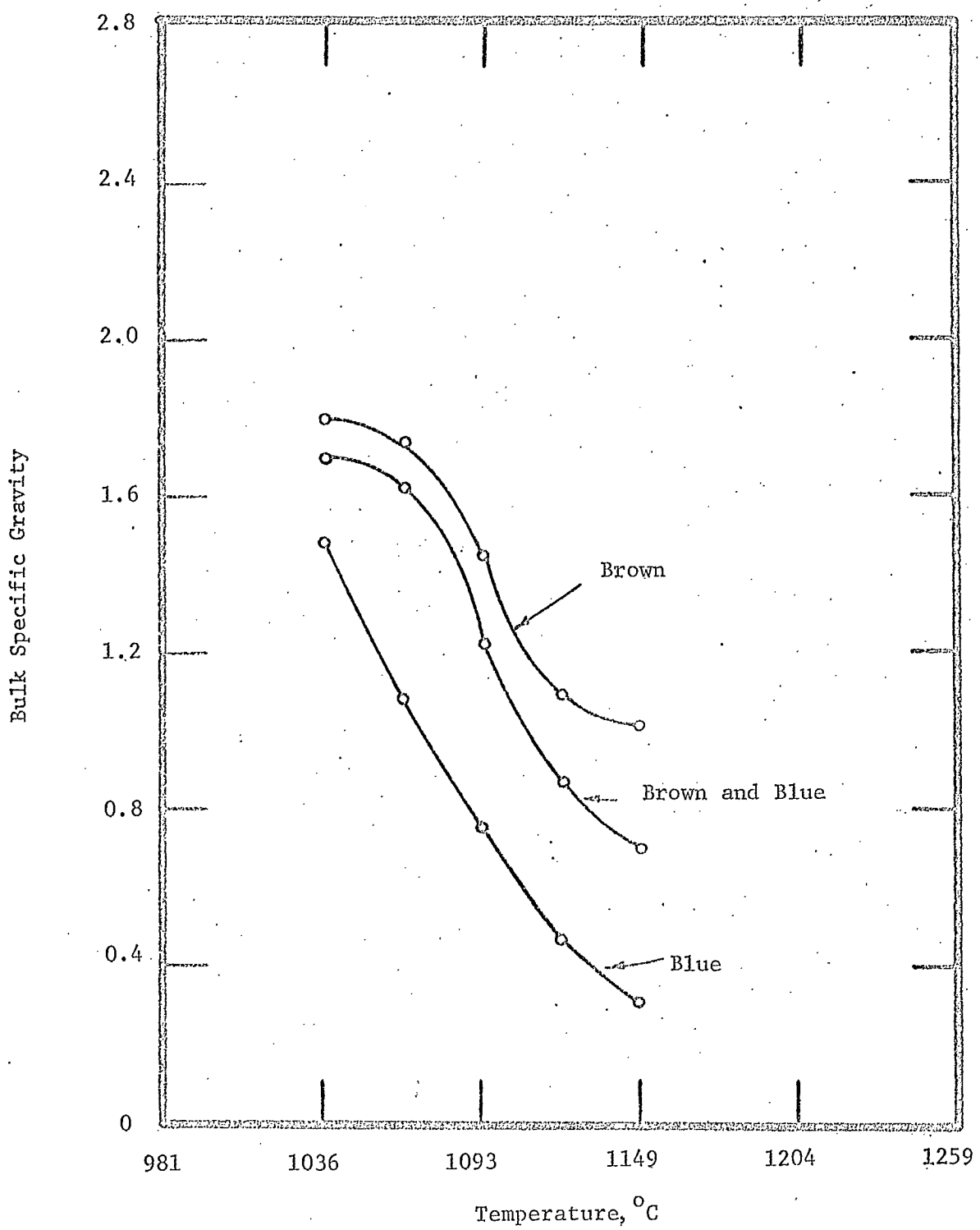


Figure 1: Stationary-kiln Tests
on Lump Shales

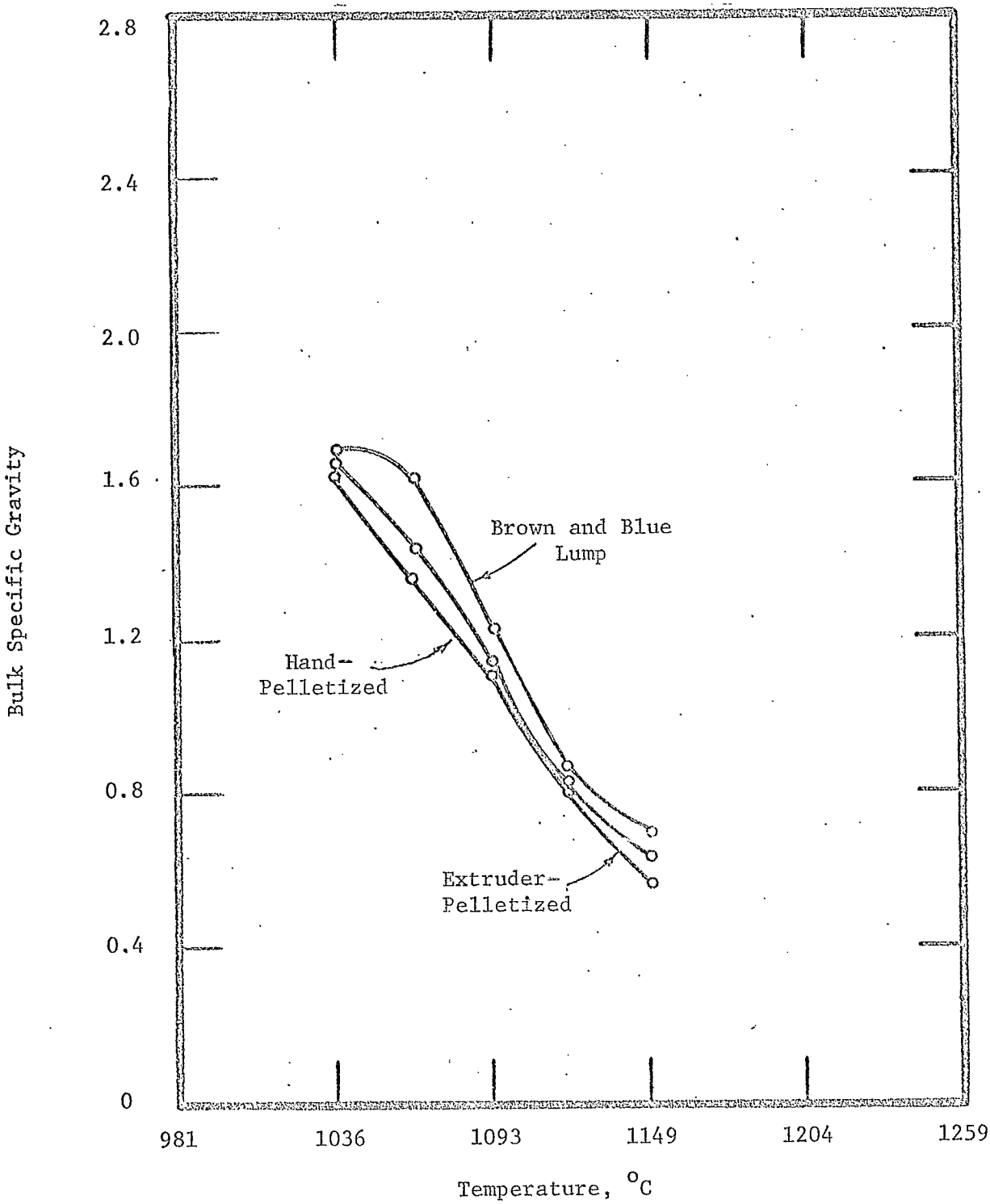


Figure 2: Stationary-kiln Tests on Combined Shales

ROTARY-KILN TESTING

Preparation of Materials

The raw material was prepared in two ways for these tests: in lump and pelletized forms.

The lump shale, including both brown and blue material, was the 1/4 to 1/2-inch fraction obtained by hand screening prior to the stationary-kiln tests. The minus 14- mesh material was brought to the plastic condition with the addition of 25 per cent water and made into cylindrically shaped pellets by means of a power-driven extrusion machine. The streams, which were 3/8 inch in diameter, were cut into lengths between 1/4 and 3/4 inches as they extruded from the die.

Both prepared materials were dried prior to being tested.

Rotary-Kiln Firing

These tests were made in a propane-fired rotary kiln, 5 inches in internal diameter and 8 feet long, set at an inclination of 1.3 in./ft and at a rotary speed of 2.6 rpm. This setting gave the material a retention time of 10 to 15 minutes, the longer retention time being at the end of the test, when the bed of material began to thin out. The burner, which had a constant gas-air ratio at all temperatures, was aimed at the lining opposite the bed of material. Impingement of the flame onto the bed itself would have resulted in localized over-heating of the surface of the particles.

For each test-firing, 1/20 cu ft (about 4 lb) of prepared material was fed to the kiln by means of a vibratory feeder, at a rate of about 10 lb/hr. Tentative firing temperatures were selected from the results of the stationary-kiln tests. The firing was begun with the temperature of the kiln lining, as measured with an optical pyrometer, about mid-way in the selected bloating-temperature range. Once the product began to discharge from the kiln, the temperature was raised quickly to that at which agglomeration of the particles began. It was then rapidly reduced to the lowest temperature at which appreciable bloating was noted. Finally, it was returned to about the mid-point between the two extremes for the balance of the firing test. About one half of the material was fired at this average temperature.

Physical Properties of Products

By measuring the volume of the product, the volume expansion of the material was determined. Each product was crushed, screened and re-combined to give the following grading:

75 per cent 3/8 in. to 4 mesh

25 per cent 4 to 8 mesh

This grading is within ASTM limits for "coarse" lightweight aggregate. The dry, loose unit weight was determined by filling a 1/30-cu ft cylindrical metal container The crushing strength of the graded aggregate was also determined. The aggregate was placed in a 3-inch-diameter steel cylinder to a depth of 5 inches. A steel plunger was placed on top of the aggregate and the aggregate was compacted 2 inches with a hydraulic press. The crushing strength is represented by two figures: the pressures required to compact the aggregate 1 inch, and a total of 2 inches.

Results

The firing temperatures and the physical properties of the product and aggregate of each test firing are shown in Table 2.

Observations

As had been anticipated from the stationary kiln tests, there was a difference in bloating characteristics between the lump shale and the pelletized shale. The firing temperature range of the lump material (about 80°F) was restricted to that at which the brown shale would bloat, it being the predominant component of the sample. With the pelletized feed, the range was lengthened from 80 to about 100°F, and no variation of bloating characteristics was evident. This better control of bloating with pelletized material resulted in a slightly greater volume expansion and lower unit weight than with the lump material. The unit weights of both aggregates were below the maximum of 55 lb/cu ft specified by ASTM Designation C330-68T "Lightweight Aggregates for Structural Concrete". The crushing strengths of the two aggregates were similar. The higher strength-weight ratio of the aggregate produced from the pelletized shale was probably due to both the shape of the particles and the uniformity of the bloating.

TABLE 2

Rotary-Kiln Tests

Feed Preparation	Firing Temperature (°C)	Average Temperature (°C)	Volume Expansion (%)	Fired Bulk Sp Gr	Unit Weight (lb/cu ft)	Crushing Strength(psi) 1"	2"
Lumps	1085-1131	1115	30	1.37	47.3	510	3020
Extruded Pellets	1069-1126	1098	40	1.14	42.2	520	2980

PETROGRAPHIC AND X-RAY ANALYSES

Petrography

Thin sections were made from specimens of the blue and brown shales to identify the coarse minerals and examine the textural relations. The sections were stained, first with alizarine red 'S' dye and then with sodium cobaltinitrite, to facilitate recognition of carbonates, quartz and feldspar.

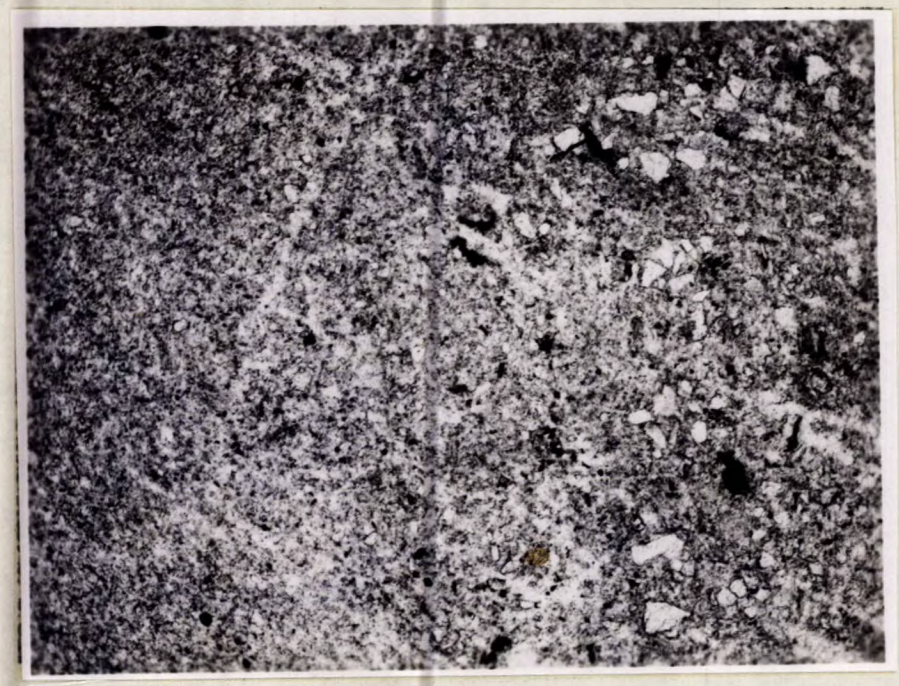
The sections indicate that the two shales are quite different petrographically. The blue rock consists predominantly of fine-grained clay minerals enclosing scattered larger fragments of quartz and feldspar (Figure 3). The brown rock is essentially a sandstone, composed mainly of angular to subrounded fragments of quartz, plagioclase feldspar, mica and altered Mg-Fe silicates, surrounded by a fine-grained brownish matrix (Figure 4); the proportions and grain sizes of fragments vary greatly in different specimens. Table 3 gives approximate mineralogical compositions of the rocks from the thin sections examined.

TABLE 3

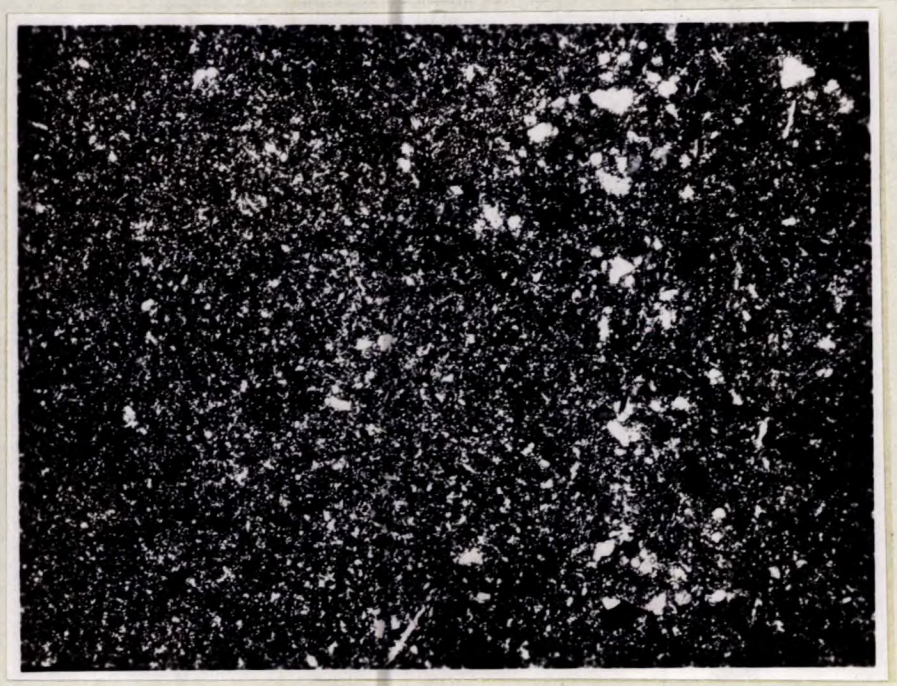
Mineralogical Compositions of the Shales

		Blue Shale	Brown Shale
(a) Thin Sections		(Section 2-69)	(Section 3-69)
	Quartz	10 %	45 %
	Plagioclase	1	10
	Calcite	-	15 *
	Mica	5	2
	Altered mafics	-	15
	Matrix (<5μ)	85 %	15 %
(b)	X-ray Diffraction (<5μ Fraction)	Montmorillonoid, Illite, Chlorite, Kaolin, Quartz, Plagioclase	Montmorillonoid, Illite, Kaolin, Quartz, Plagioclase

* Much less in other specimens.



A. Polarized Light



B. Cross-Polarized Light

Figure 3. Photomicrographs of blue shale, showing scattered, large fragments of quartz and (rare) feldspar in a fine-grained matrix of clay minerals. Magnification 50X.

Figure 4. Photomicrograph of brown 'shale', showing abundant fragments of quartz, feldspar (white), altered mafics (pale grey, e.g. A) and calcite (dark grey), imbedded in opague brownish matrix (black). Polarized light; magnification 50X.

X-Ray Diffraction Analyses

Mineralogical compositions of the fine fractions of the shales were determined by X-ray diffraction analysis, using the procedures outlined by Brindley (1) and Warshaw and Roy (2).

Coarsely pulverized (1-2 mm) rock was agitated in demineralized water by magnetic stirrer for several hours to free clay particles, and the plus 5-micron fraction was separated by centrifugation. A portion of the minus 5-micron suspended clay was sedimented onto a borosilicate glass slide and scanned with a Norelco High-Angle X-ray diffractometer in three states: (a) untreated, air-dried; (b) saturated with ethylene glycol; and (c) heat treated at 580°C for 1/2 hour. Another portion of the suspension was centrifuged to remove excess water, and the sediment was boiled for 1/2 hour in concentrated hydrochloric acid. After successive washings and centrifugation in demineralized water, the residue was sedimented onto

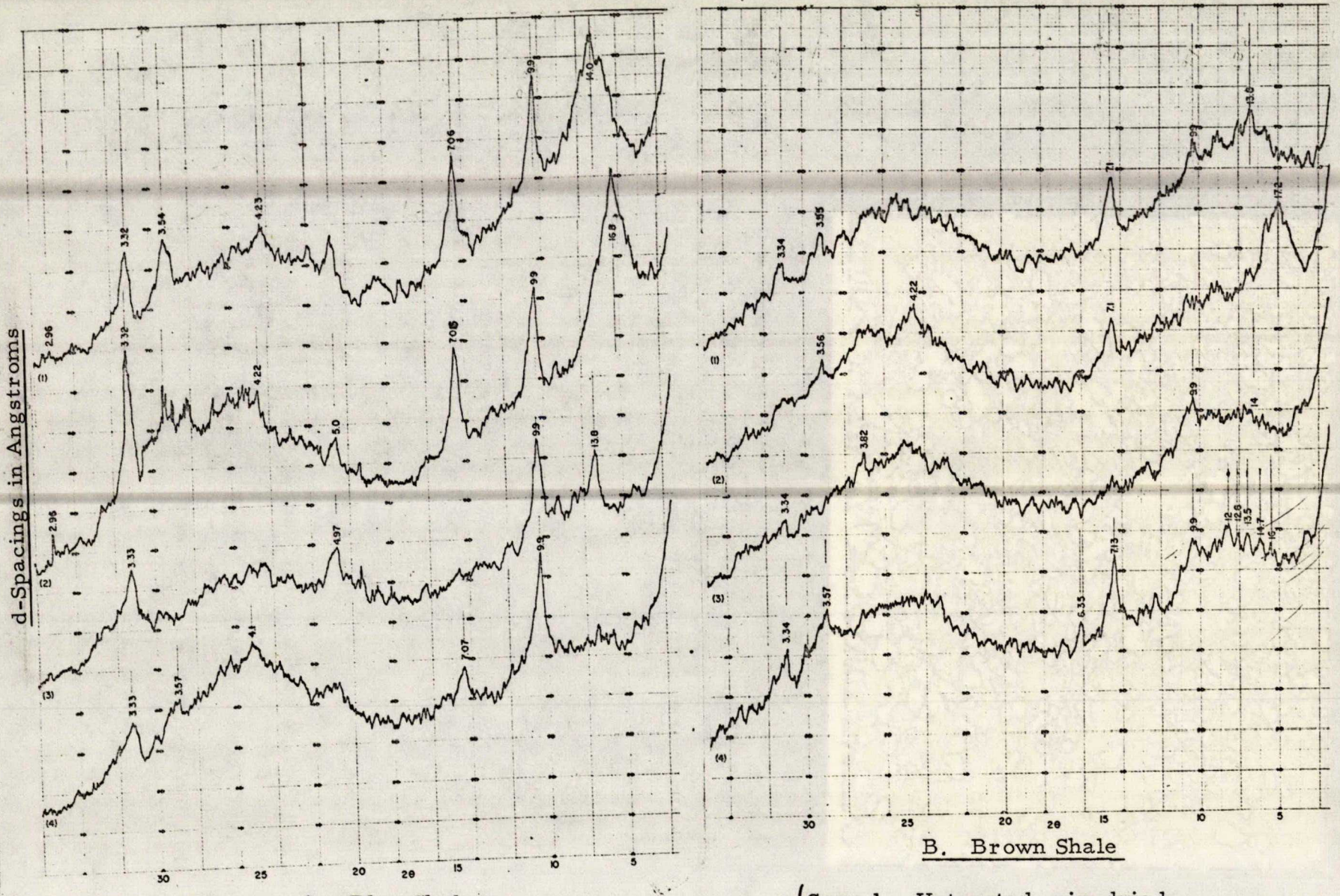


Figure 5. X-ray diffractograms of oriented mounts of <5-micron fractions of blue and brown Taber shales.

Scan 1. Untreated, air-dried
Scan 2. Saturated with ethylene glycol
Scan 3. Heated 1/2 hr at 580°C
Scan 4. Boiled in conc. HCl

a glass plate and analyzed in the X-ray diffractometer. Unoriented mounts of the untreated dried clays were also analyzed in a Guinier-deWolff powder diffraction camera.

The results of the diffractometer analyses are compared in Figure 5, and summarized in Table 3(b). The principal clay minerals in the blue shale are a montmorillonoid, illite (mica), chlorite and kaolin, with quartz and minor plagioclase. The same minerals, with the exception of chlorite, are present in different proportions in the brown shale; the Guinier diffraction pattern shows that it contains a little goethite as well.

The montmorillonoid is abundant in both rocks, as shown by development of a prominent $\pm$ 17 Å peak upon glycolation and its collapse upon heating. Chlorite is fairly abundant in the blue shale but not in the brown because a 13.8Å reflection which developed in the former with heat treatment, and vanished with hydrochloric acid treatment, did not appear in the latter. Illite is also much more abundant in the blue shale, according to the relative peak heights of the basal 10 Å reflection. Kaolin, however, is plentiful only in the brown shale, as is indicated by the prominent basal reflections at 7 Å and 3.5 Å which disappear upon heating.

DISCUSSION

The mechanism of bloating of rocks by firing is well known (3, 4, 5), the principal requirements being the formation of a viscous glass on the surface and within a particle, which prevents escape of gases formed later in the body of the particle. To effect bloating, therefore, the raw material must contain a phase which fuses easily or reduces the melting point of refractory minerals, and a phase which evolves a gas upon heating.

The Taber shales meet both requirements. They contain hydrated minerals (montmorillonoid, illite, and calcite in the brown shale), which provide water vapor and carbon dioxide for the bloating process; and they contain iron, at least partly in the ferrous state in illite or montmorillonoid, which would cause fluxing of the more refractory minerals (kaolin, quartz) during firing to produce the necessary viscous melt. The degree of bloating would depend mostly upon the proportions of these fusible minerals present. Therefore the blue shale, with its high content of clay minerals, bloats well whereas the sandy, more refractory brown shale bloats relatively poorly.

CONCLUSIONS

The sample submitted was composed of two shales that differ widely in petrography and bloating characteristics. The blue shale, which comprised about 10 per cent of the sample, contained a higher proportion of easily fusible minerals, and bloated much better than did the brown shale.

If processed in lump form, a mixture of these shales would not make an acceptable lightweight aggregate. There would be too wide a variation of specific gravity of the resultant particles, which would create a severe segregation problem. The shales should be processed separately. Either shale by itself would probably give an acceptable lightweight aggregate. The blue shale would bloat at a lower temperature, through a wider temperature range, and it would yield an aggregate of lower unit weight than the one produced from the brown shale.

Grinding and pelletizing the mixture of the two shales would eliminate non-uniformity of bloating and yield an acceptable lightweight aggregate. The bloating temperature range and the unit weight of the resulting lightweight aggregate would fall between those values for the two shales processed singly.

ACKNOWLEDGEMENTS

We wish to express our appreciation to Dr. R.S. Dean for his helpful comments in the study of the clay mineralogy, and to Messrs. H. Childe and M. Raicevic for their assistance in the laboratory work.

REFERENCES

1. Brindley G.W., "Identification of Clay Minerals by X-ray Diffraction Analysis"; In: Clays and Clay Technology, California Dept. of Nat. Res. Bulletin 169, 119-129 (1955).

2. Warshaw C.M., Roy R., "Classification and a Scheme for the Identification of Layer Silicates"; Bull. Geol. Soc. Amer. 72, 1455-1495 (1961).

3. Vrublevskii L.E., "On the Causes of Bloating of Clay Rocks"; Glass and Ceramics (Russian Transl.), v.19(1), 22-24 (1962).

4. Riley C.M., "Relation of Chemical Properties to the Bloating of Clays", Journ. Amer. Ceramic Soc. 34(4), 121-128 (1951).

5. Hill R.D., Crook D.N., "Some Causes of Bloating in Expanded Clay and Shale Aggregates", Australian Journ. App. Sc., 11(3), 374-384 (1960).

HSW/JAS:jh