COMPARATIVE PROPERTIES OF AGGREGATES FOR SURFACE TREATMENTS CONSIDERED FOR USE IN DISTRICT 1

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TEXAS HIGHWAY DEPARTMENT

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by

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Texas Highway Department

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PREFACE

This report is the result of laboratory and field studies made on seventeen aggregate materials, sixteen of which have been used in certain paving operations on highway projects in District 1 and one a potential source. The study originated from a specific request from District 1 personnel for the Materials and Tests Division to provide assistance in evaluating aggregate materials, specifically those considered for use in surface treatments, presently available to the District so that, District personnel in turn, can plan future use of these materials which take both economic factors and physical properties into account.

This study is a specialized part of an overall comprehensive reference study of geological and engineering properties of existing aggregate sources used throughout the state.

CONTENTS

I. SUBJECT

A critical loss of skid resistance exists with the use of certain aggregates on surface treatments. The presentation of several comparative properties of the various surfacing aggregates currently being considered for use in District 1 is the subject of this study.

II. PURPOSE

The purpose of this report is to show interrelationships between certain measurable parameters and the desired engineering requisites of a number of coarse aggregate materials. These findings were obtained from laboratory examinations and field studies on materials currently being used or considered by District 1 personnel.

III. SUMMARY AND CONCLUSIONS

1. Laboratory and field studies on seventeen aggregate materials have been conducted in order that any relationships between various measurable properties may be better understood.
2. The aggregate types considered for this study have been compared with respect to petrographic characteristics, polishing features as measured by the British Portable Tester and the skid trailer, Los Angeles Abrasion, and insoluble residue analysis.
3. Petrographic analyses indicate that composition and rock fabrication appear to be related to frictional characteristics of an aggregate as measured by the British Portable Tester.
4. In terms of polish value, the vesicular synthetic aggregates had the highest determined values (51-62); impure dolomites,

impure limestones and "trap rock" had the next highest values (42-50); the relatively pure limestones, dolomites and siliceous river gravels had the lowest polish values (30-39).

5. In general, skid-trailer readings at 40 m.p.h. taken on surface treatments having greater than one million traffic applications reflect the same polishing characteristics as shown by the polish value test.
6. Correlations between the Los Angeles Abrasion Test and the observed polishing features of an aggregate as determined by polish value tests or skid-trailer data was not found in this study.
7. Insoluble residue analyses on carbonate aggregates, in general, suggest that the greater the insoluble residue, the better chance it has to resist polishing.

IV. MATERIALS AND METHODS

A. Location and Geologic Setting of Aggregate Sources.

Generalized locations for the seventeen sources considered in this study are plotted on a County Outline Map and illustrated as Figure 1. More detailed maps for sources located in Wise, Eastland and Uvalde Counties are in Appendix I.

The three Oklahoma materials sources currently being used by District 1 and included in this report are located as follows:

Trinity Concrete Products - Stringtown Plant: Located at the northeastern edge of the village of Stringtown, Atoka County, Oklahoma. The large open-pit quarry, which lies just east of

LOCATION OF COARSE AGGREGATE SOURCES

CONSIDERED FOR USE IN DISTRICT 1

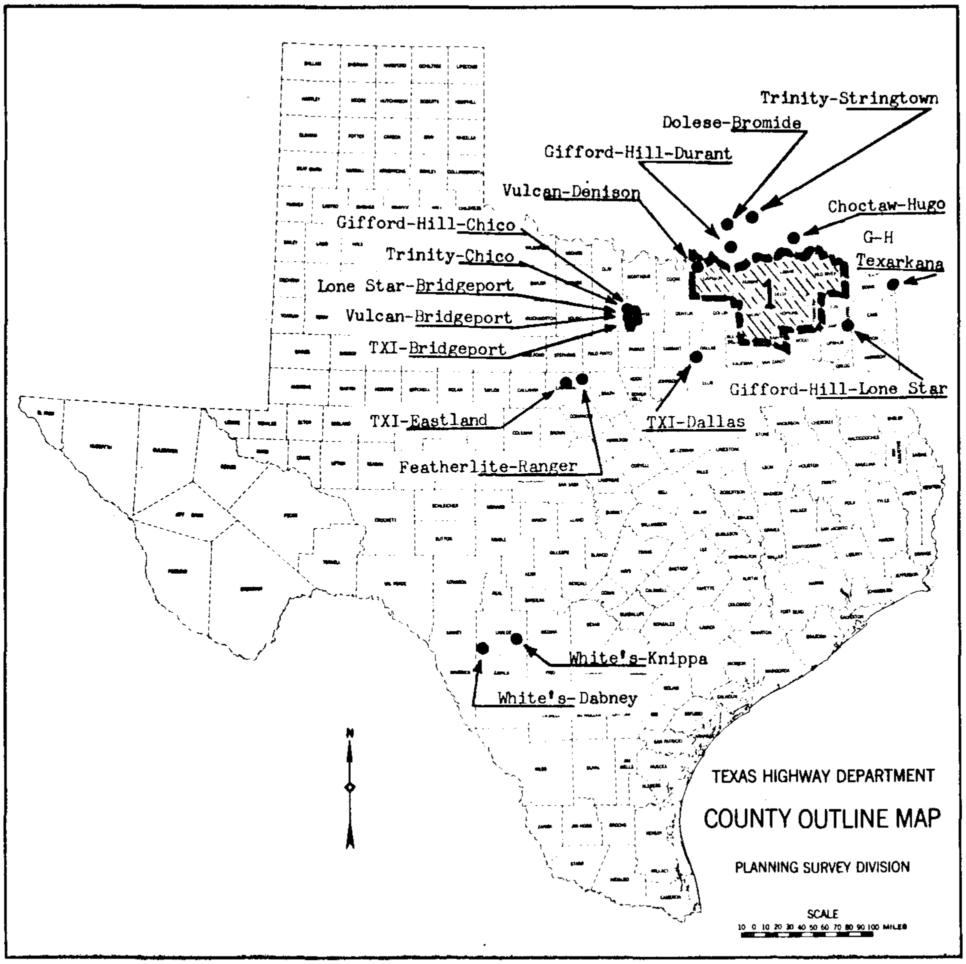


Figure 1

the Kansas and Texas R. R., is situated in the vertically dipping, thin-bedded Wapanucka Formation, a prominent ridge-forming series of rock units on the western flank of the Ouachita Mountain complex. Production mainly consists of cherty dolomites.

Dolese Brothers - Bromide Plant: Located just out of the town of Bromide, in the southwestern corner of Coal County, Oklahoma. The quarry lies in a faulted complex of interbedded siliceous limestones and dolomites on the northeastern flank of the Arbuckle Mountains. Lithologic characteristics appear to be similar to the Wapanucka Formation.

Choctaw Materials Company - Hugo Plant: Located some 4 miles east of Hugo, Choctaw County, Oklahoma. Plant operations mainly consist of stripping residual gravels from terrace and floodplain deposits on the edge of the Kiamichi and Red River meander belts. The materials mainly consist of well-rounded brown and yellowish-colored siliceous and limestone gravels.

A recently acquired source located about 20 miles north of Durant, Bryan County, Oklahoma, has been opened by Gifford-Hill and Company. This potential source consists of a fine-grained light to dark gray-colored dolomite and is probably associated with the Arbuckle Mountain complex.

The only coarse aggregate materials source investigated for this report located within District 1 is the Vulcan Materials, Denison plant. Formerly Crusher's, Inc. (Wible Pit), the source is located some 10 miles northwest of Denison, north of Fink, near Lake Texoma. Geologically, the material is being quarried from the Goodland Limestone Formation, which mainly consists of medium-bedded, cream to gray-colored marly and carbonaceous limestone.

Other Texas sources currently being utilized by District 1 and located outside district boundaries are as follows:

<u>Gifford-Hill, Texarkana</u>: The currently active source operated by Gifford-Hill at Texarkana is located 10 miles south of town near the community of Hoot in southern Bowie County. The source, Hoot Pit, is geologically situated on the northern edge of the Sulphur River floodplain just off FM 558. There are several other local sources near-by. The production is essentially a multi-colored siliceous gravel; however the material from this area is characterized by its distinct red jasper (chert) particles.

<u>Gifford-Hill, Lone Star</u>: The Pete Plant of Gifford-Hill and Company is located about 1 mile south of Lone Star on U.S. 259 in southern Morris County. The aggregate source utilizes an iron blast furnace slag (often referred to as Daingerfield slag) produced as a waste product from the Lone Star Steel Company. The material consists of glassy, often crystalline, synthetic chemical compounds which are generally gray in color and, although it has a conventional bulk unit weight, it is vesicular.

Gifford-Hill, Perch Hill Plant, Chico: The plant and open-pit quarry are located about 2 miles south and 1 mile east of Chico, Wise County. The material consists of a white to cream colored fossiliferous limestone, geologically known as the Chico Ridge Limestone formations (Pennsylvanian Age).

Trinity Concrete Products, Chico: This source (formerly Southwest Stone) is located adjacent to and just north of the above listed site some 1 mile south and 1 mile east of Chico. Production is also from the Chico Limestone formation.

Vulcan Materials Company, Bridgeport: Located 1 mile west of SH 114 about 5 miles north of Bridgeport, Wise County. This source (formerly Crusher's, Inc.) is due west of the Gifford-Hill plant and produces crushed stone from the Chico Limestone formation.

Texas Industries, Inc., Bridgeport: The processing plant of TXI is located just west of SH 114 about 5 miles north of Bridgeport and the open pit quarry is some 2 miles west of the plant. The crushed stone is from the Chico Limestone formation.

Lone Star (North Texas Agcrete), Bridgeport: The pit and plant site (formerly Wesco) are located about 5 miles northwest of Bridgeport off FM 1658 near Lake Bridgeport. The crushed stone is from the same formation noted above.

Texas Industries, Inc. (Dallas Lightweight Aggregate Company), Dallas: The TXI-Dallas processing plant is located at Eagle Ford on Chalk Hill Road at Dallas-Fort Worth Turnpike, about 1 mile east of Loop 12 in Dallas County. The operation consists of firing in rotary kilns a raw shale quarried from an open pit located about 1/2 mile southwest of the plant area. Geologically, the shale is from the Eagle Ford formation (Upper Cretaceous Age).

Featherlite Corporation, Ranger: The plant is located on the northern city limits of Ranger in Eastland County. The shale for processing comes from two open pits; one just to the north of the plant area, and the other about 5 miles south of town. The shale is from geological units called the Seaman Ranch beds, Brad formation (Pennsylvanian Age).

Texas Industries, Inc. (Texas Lightweight Aggregate Company), Eastland: The TXI-Eastland plant is located at the northwest edge of town on the Lake Brelsford road. The shale pit is located about 3 miles south of the plant and 1/2 mile southwest of the IH 20-SH 6 intersection. The pit is within the Finis shale unit, Graham formation (Pennsylvanian Age).

White's Mines, Inc., Knippa: Formerly the old Southwest Stone plant and more recently Trinity Concrete Products, the Knippa plant of White's Mines is located 1/2 mile west of town on U.S. 90 at the Dry Frio River. The crushed stone consists of a very dense, dark gray to black basalt ("Trap Rock") which occurs as igneous intrusions (volcanoes) in numerous sites throughout Uvalde County.

White's Mines, Inc., Dabney: The samples of limestone rock asphalt examined for this report were sampled from stockpiles at the White's Mine Dabney plant but can be considered equivalent to the rock asphalt produced by Uvalde Rock Asphalt Company at Blewett. Both sources, located 15 miles west and 6 miles south of Uvalde on FM 1022, are situated in the asphalt-bearing Anacacho Limestone (Upper Cretaceous Age).

B. Aggregate Samples.

Samples from four of the seventeen sources treated in this study were collected and submitted by District 1 personnel. The remaining samples were taken by personnel of the Materials and Tests Division. These samples represent production material destined for Texas Highway projects and were taken by standard sampling procedures. After the samples were received by the laboratory, they were screened, washed and dried in preparation for testing.

C. Petrographic Analysis.

Representative aggregate particles were taken from the material prepared for the polish value test. The particles were selected on the basis of color, texture and other gross lithologic characters in order to get a wide spectrum of petrographic variation or degree of heterogeneity. Several thin sections were prepared using standard petrographic techniques which involved cutting, mounting on glass microscope slides, and grinding to about 30 microns in thickness. The rock specimen, after proper thickness was achieved, was then viewed at magnifications up to 1250X utilizing transmitted polarized light. Mineral compositions were determined by their respective optical properties and crystalline structures.

D. Polish Value Determinations.

Aggregate particles, graded to pass 1/2" and retained on #4 screen, were selected at random, placed in special 3-1/4" - 2-1/4" steel molds, and molded with a polyester resin. A minimum of seven molded specimens were tested for a given source. After removal, the molded specimens were tested for an initial friction reading utilizing the British Portable Tester (BPT). Each was then mounted on the British Accelerated Polishing Machine, which holds 14 specimens at a time, and run for a minimum of 9 hours (approximately 180,000 cycles). Silicon carbide abrasive grit #150 and water were applied between an inflated rubber tire and the specimens to accelerate the wearing and/or polishing action. After the prescribed running time, the specimens were again tested with the portable tester to obtain a final frictional reading designated as the polish value.

E. Skid Trailer Readings.

Skid trailer studies had previously been conducted by Highway Design Research personnel on pavements in several Districts containing 11 of the subject aggregate materials. Data on both hot-mix asphaltic concrete (HMAC) and surface treatments (S.T.), were obtained in order that comparisons could be made with other properties of the aggregates such as polish value. Trailer readings were taken when the paving material was first put into service and follow-up readings were subsequently taken to find out how the aggregates polish under traffic. Readings were taken as the trailer was towed at 40 m.p.h. A mechanical device, activated just before the trailer brakes are applied, feeds water onto the pavement in front of the tire.

F. Los Angeles Abrasion Tests.

Los Angeles Abrasion test results for 15 of the 17 subject aggregate sources were taken from Materials and Tests Division files. The number of tests represented ranged from 6 to 18 and cover a span of 5-6 years. The readings, taken from "A," "B" and "C" wears, were tabulated and averaged. The abrasion test was not considered for the limestone rock asphalt sample, although there is a specification value based on the white rock. The Gifford-Hill source north of Durant, Oklahoma, has not been submitted for a project to date and the preliminary sample (taken from test cores) submitted for polish value was inadequate in quantity for other tests to be performed.

G. Insoluble Residue Analyses.

Nine of the subject aggregate materials were chemically analyzed for their acid insoluble residue content. The non-carbonate types (Hugo, Texarkana, Knippa and the synthetics) were excluded from this test.

For the rock asphalt sample, the asphalt was extracted from the sample and acid insolubles were determined on the remaining carbonate fraction (essentially a fragmented fossiliferous limestone debris).

The procedure used for analyses of total acid insolubles is outlined in Appendix II.

V. RESULTS

Data collected from polish value determinations, initial frictional readings (I.R.) and the percent loss or gain in frictional properties (± % Polish), Los Angeles Abrasion Tests, along with a brief rock type classification of the seventeen aggregate sources examined for this study are summarized in Table I.

A graph which illustrates the comparative relationships between the initial frictional reading and the final polish values of each of the aggregate sources is shown in Figure 2. As can be seen, the sources are ranked according to decreasing polish values. The initial frictional readings have been plotted to illustrate relative degrees of polishing. The graph shows quite well that crushed aggregates, regardless of composition, have a relatively high initial reading (between 50 and 60), whereas, well worn river gravels have a low initial reading in addition to a low final value.

Another polishing parameter taken from skid trailer readings has been plotted against polish values and illustrated in Figure 3. The values taken from the skid trailer (Skid number at 40 m.p.h. -SN_{40})

TABLE I

SOURCE	ROCK TYPE	POLISH VALUE	I.R.	% POLISH	L.A. ABRASION
TXI - Dallas	Expanded Shale	62	57	+9	21 (7)*
Featherlite - Ranger	Expanded Shale	58	57	+2	21 (8)
Gifford-Hill - Lone Star	Blast Furnace Slag	52	59	-12	36 (18)
TXI - Eastland	Expanded Shale	51	52	-2	20 (12)
White's - Uvalde	Limestone Rock Asphalt	50	60	-17	**
Trinity - Stringtown	Cherty-Dolomite	46	57	-19	20 (24)
Dolese - Bromide	Cherty-Dolomitic Limestone	45	56	-20	26 (12)
White's - Knippa	"Trap Rock" Basalt	44	51	-14	14 (10)
Vulcan - Denison	Argillaceous Limestone	42	59	-22	37 (14)
TXI - Bridgeport	Limestone	39	59	-34	28 (15)
Lone Star - Bridgeport	Limestone	37	57	-35	25 (9)
Trinity - Chico	Limestone	35	57	-38	24 (18)
Vulcan - Bridgeport	Limestone	35	58	-40	26 (9)
Gifford-Hill - Chico	Limestone	34	57	-40	25 (15)
Gifford-Hill - Durant	Dolomite	34	50	-32	**
Choctaw - Hugo	Siliceous Gravel	31	32	-3	21 (6)
Gifford-Hill - Texarkana	Siliceous Gravel	30	32	-6	23 (15)

* Number of Abrasion Tests.
** Data Unavailable.
I.R. = Initial Reading

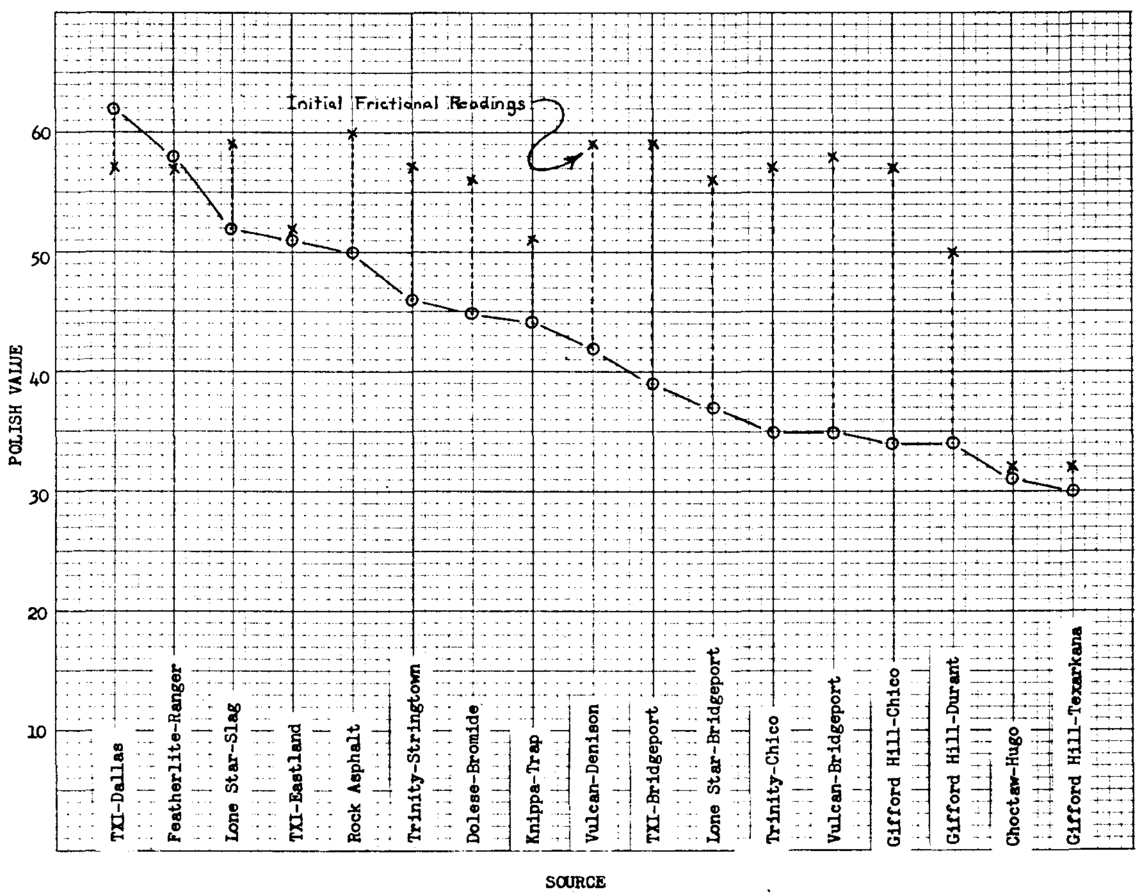

POLISH VALUE
10
20
30
40
50
60
Initial Frictional Readings
TXI-Dallas
Featherlite-Ranger
Lone Star-Slag
TXI-Eastland
Rock Asphalt
Trinity-Stringtown
Dolese-Bromide
Knippa-Trap
Vulcan-Denison
TXI-Bridgeport
Lone Star-Bridgeport
Trinity-Chico
Vulcan-Bridgeport
Gifford Hill-Chico
Gifford Hill-Durant
Choctaw-Hugo
Gifford Hill-Texarkana
SOURCE

Figure 2

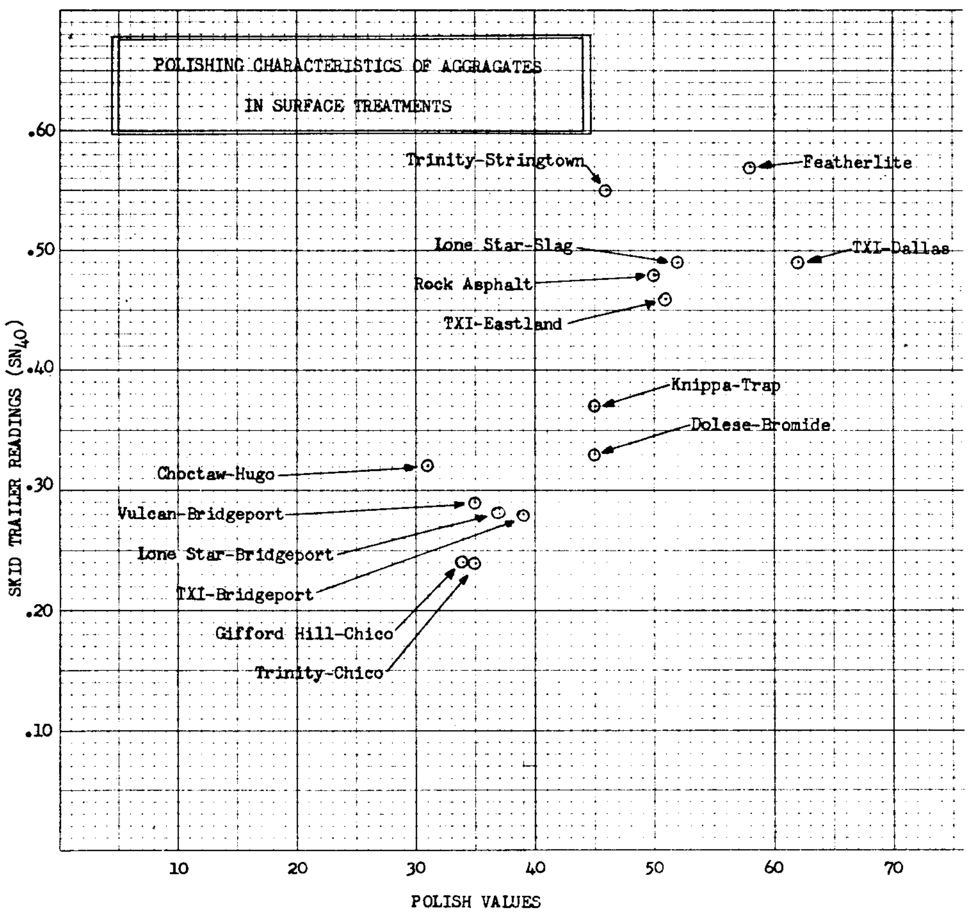

POLISHING CHARACTERISTICS OF AGGRAGATES
IN SURFACE TREATMENTS
Trinity-Stringtown
Featherlite
Lone Star-Slag
TXI-Dallas
Rock Asphalt
TXI-Eastland
Knippa-Trap
Dolese-Bromide
Choctaw-Hugo
Vulcan-Bridgeport
Lone Star-Bridgeport
TXI-Bridgeport
Gifford Hill-Chico
Trinity-Chico
.60
.50
.40
.30
.20
.10
10
20
30
40
50
60
70
SKID TRAILER READINGS (SN_{40})
POLISH VALUES

Figure 3

need to be qualified when attempting to make any correlation with polish value readings. The skid numbers, for several of the sources, as plotted in Figure 3, represent an average of several trailer readings taken for a given source and are listed in Table II. These trailer readings were counted if the test strip had been subjected to more than one million traffic applications. Skid trailer data indicated that, in general, the subject crushed aggregate materials, regardless of composition, started out high and fell in readings as the number of vehicle applications increased. Also, the higher the average daily traffic (ADT), the faster some materials would polish, however, the composition of the aggregate had considerable effect on the rate of polish.

The analysis of acid insoluble constituents for nine of the subject sources (those which contain carbonates) is listed in Table III. In general, those with greater amounts of insoluble residues had somewhat higher indexes of polish values (P.V.) and skid trailer readings (SN_{40}). The average polish values and skid trailer numbers are listed for comparison. It appears that in order for a carbonate rock to have a long lasting and acceptable skid resistance as indicated by the skid trailer, the insoluble residue content has to be relatively high. The lack of an adequate number of samples makes any clear-cut relationships between these two parameters inconclusive at this point.

T A B L E I I

Source	Skid No. 40 mph*	No. Readings	District
Featherlite - Ranger	57	6	14
Trinity - Stringtown	55	2	1
TXI - Dallas	49	2	10
Lone Star Slag	49	20	19
Rock Asphalt	48	7	15
TXI - Eastland	46	3	23
Knippa Trap Rock	37	16	15
Dolese - Bromide	33	5	1
Choctaw - Hugo	32	2	1
Vulcan - Bridgeport	29	1	1
Lone Star - Bridgeport	28	1	1
TXI - Bridgeport	28	5	1
Gifford-Hill - Chico	24	27	1
Trinity - Chico	24	14	1

* Average of readings on pavements having greater than 1 million traffic applications (some had as high as 20 million).

Data supplied by Highway Design Division.

TABLE III

Source	Acid Insolubles (%)	P.V.	SN_{40}
Trinity - Stringtown	58.5	46	.55
Dolese - Bromide	12.0	45	.33
Rock Asphalt - Dabney	4.0*	50	.48
Lone Star - Bridgeport	3.4	37	.28
Vulcan - Denison	2.2	42	**
TXI - Bridgeport	2.0	35	.28
Vulcan - Bridgeport	1.8	39	.29
Gifford-Hill - Chico	1.5	34	.24
Trinity - Chico	1.2	35	.24

*Does not include asphalt content

**Data unavailable

VI. DISCUSSION

The engineering requisites of aggregates is a matter of much concern because of the increasing requirements imposed by high volumes of traffic and the corresponding rapid rate of polish of pavement surfaces affected by this traffic. This is of particular concern where the pavement type is a seal coat or surface treatment and the exposed aggregate is the component which primarily contributes to the skid resistance provided by that surface.

Data presented in this study clearly indicates that techniques are available which enable trained personnel, responsible for making decisions concerning the use of aggregate materials, to effectively anticipate a material's susceptibility to traffic polishing. Field experience has shown that there is a difference in skid-resistance performance of various aggregate types. Laboratory studies, which incorporate several means of examining the various properties of an aggregate, can aid in establishing a range of physical and chemical parameters and therefore assist in predetermining an aggregate's potential as related to polishing rate and skid resistance.

Petrographic analyses on the materials considered for this study indicate that composition and rock fabrication (how the mineral constituents are arranged) appear to be related to frictional characteristics of an aggregate as measured by the British Portable Tester. Microscopic examination of aggregate surfaces, after exposure to wear and accelerated polishing in the laboratory, reveals that particle microtexture (or the lack of it) appears to be related to the polishing characteristics of the particle. In Table I, the seventeen sources are ranked in descending order according to polish value, and correspondingly, their respective surface microtextures (as viewed microscopically) appear to be ranked.

The aggregate types with the greatest polish value are the vesicular synthetic materials (the expanded shales and slags) followed by impure carbonate types, except for the basalt sample, then the relatively pure carbonates, and lastly, the predominantly siliceous and well rounded (water polished) river gravels. Studies conducted by the Materials and Tests Division on well indurated sandstone aggregates (Duval and Freestone Counties) indicate that certain sandstones have polish values which rank as high as vesicular synthetic aggregates because of their gritty microtexture.

Correlation with our standard Los Angeles Abrasion Tests and results of polishing tests (as measured by polish value or skid trailer) has not been found in this study. For example, the expanded shale aggregate produced at Dallas had a high polish value reading and a fairly low average Los Angeles wear, but the samples being tested showed an appreciable amount of particle attrition after 9 hours of accelerated polishing. Its conceivable that after a few additional hours of polishing the particles would disintegrate although there would be no significant loss in polish value. Therefore, polish values alone should not be the only parameter considered in aggregate evaluation. The Los Angeles Abrasion results listed in Table I fail to show any relationship when compared to corresponding polish values.

Although there are exceptions, the rate of polish (expressed as percent loss in frictional values) is inversely related to polish value. Figure 2 illustrates nicely this inverse relationship. The figure also shows that crushed aggregates, regardless of type, have relatively high initial frictional readings, whereas, the river gravels start out low and end up low.

The skid trailer data listed in Table II has been plotted against corresponding polish values for 14 of the examined sources. Figure 3 illustrates the comparison between polishing parameters as measured by two different methods. As shown, those aggregate types having high polish values have correspondingly high skid-trailer readings (average reading after exposure to greater than one million vehicle applications). It should be noted when considering comparisons such as that shown in Figure 3 that, although an aggregate type appears acceptable in terms of its polish value, a surface treatment in a flushed condition will generally show a low skid-trailer reading.

To provide an additional insight for comparing polishing characteristics of surfacing aggregates, skid-trailer readings have been plotted against total traffic for surfaces with four of the aggregate types and illustrated as Figure 4. The data includes readings for two synthetic materials, a "Trap Rock" and the Wise County limestones which shows their respective polishing characteristics as measured

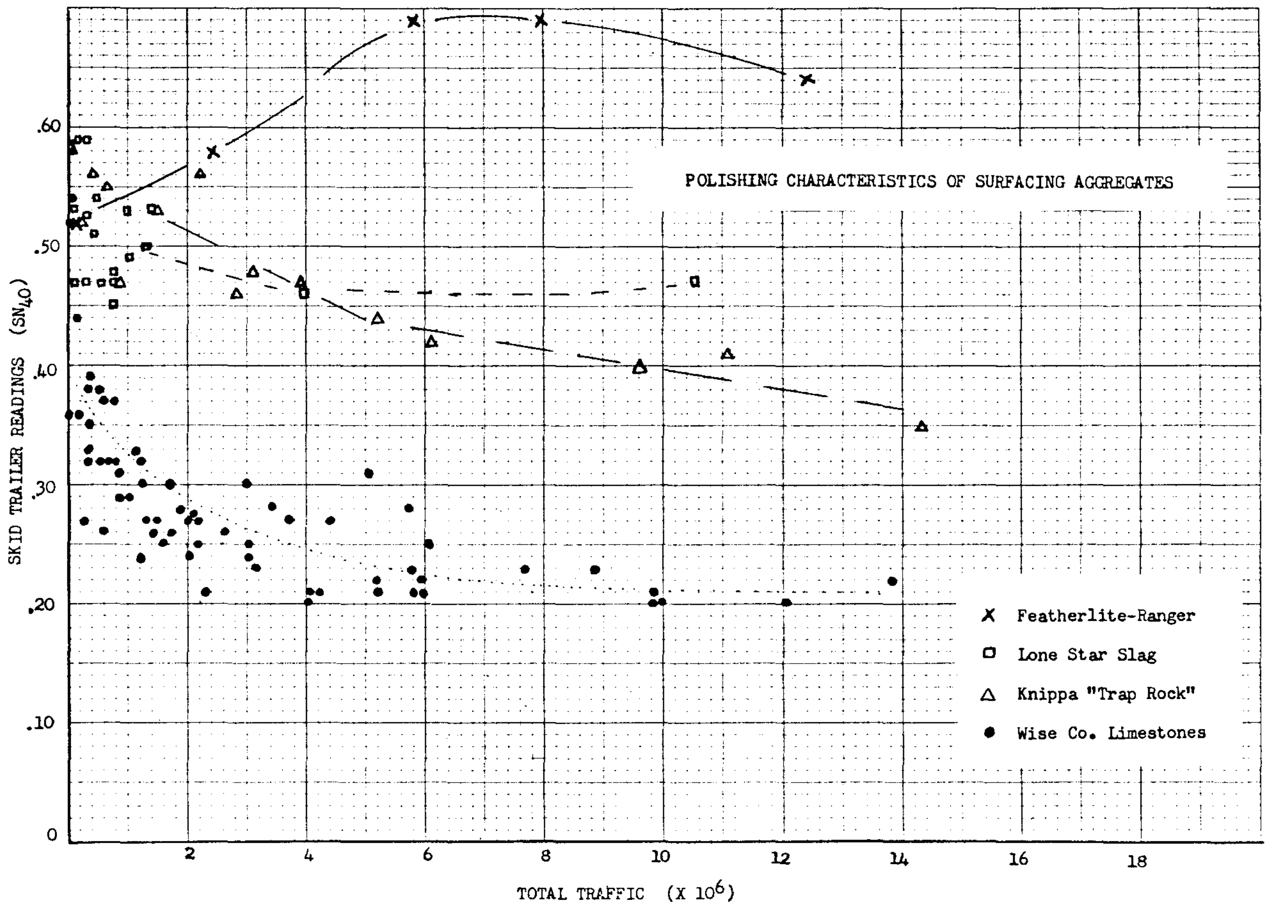
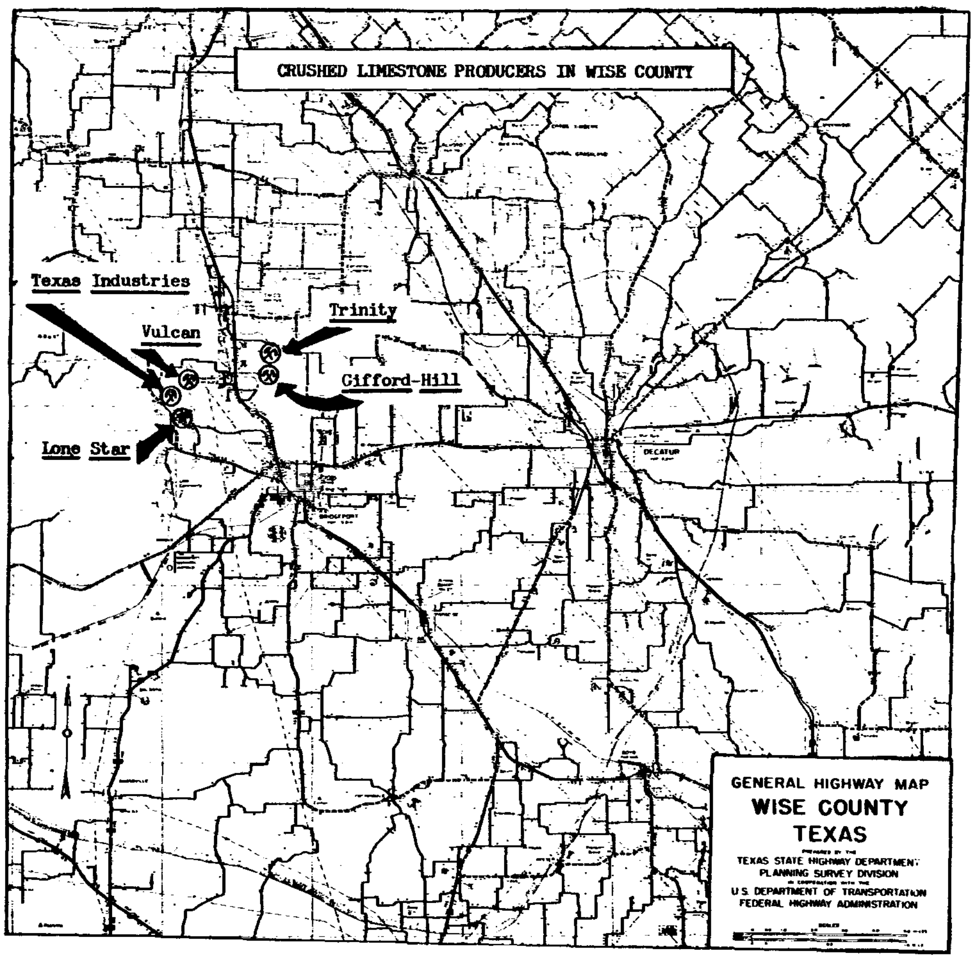

POLISHING CHARACTERISTICS OF SURFACING AGGREGATES
SKID TRAILER READINGS (SN_{40})
TOTAL TRAFFIC (X 10^6)
.60
.50
.40
.30
.20
.10
0
2
4
6
8
10
12
14
16
18
Featherlite-Ranger
Lone Star Slag
Knippa "Trap Rock"
Wise Co. Limestones

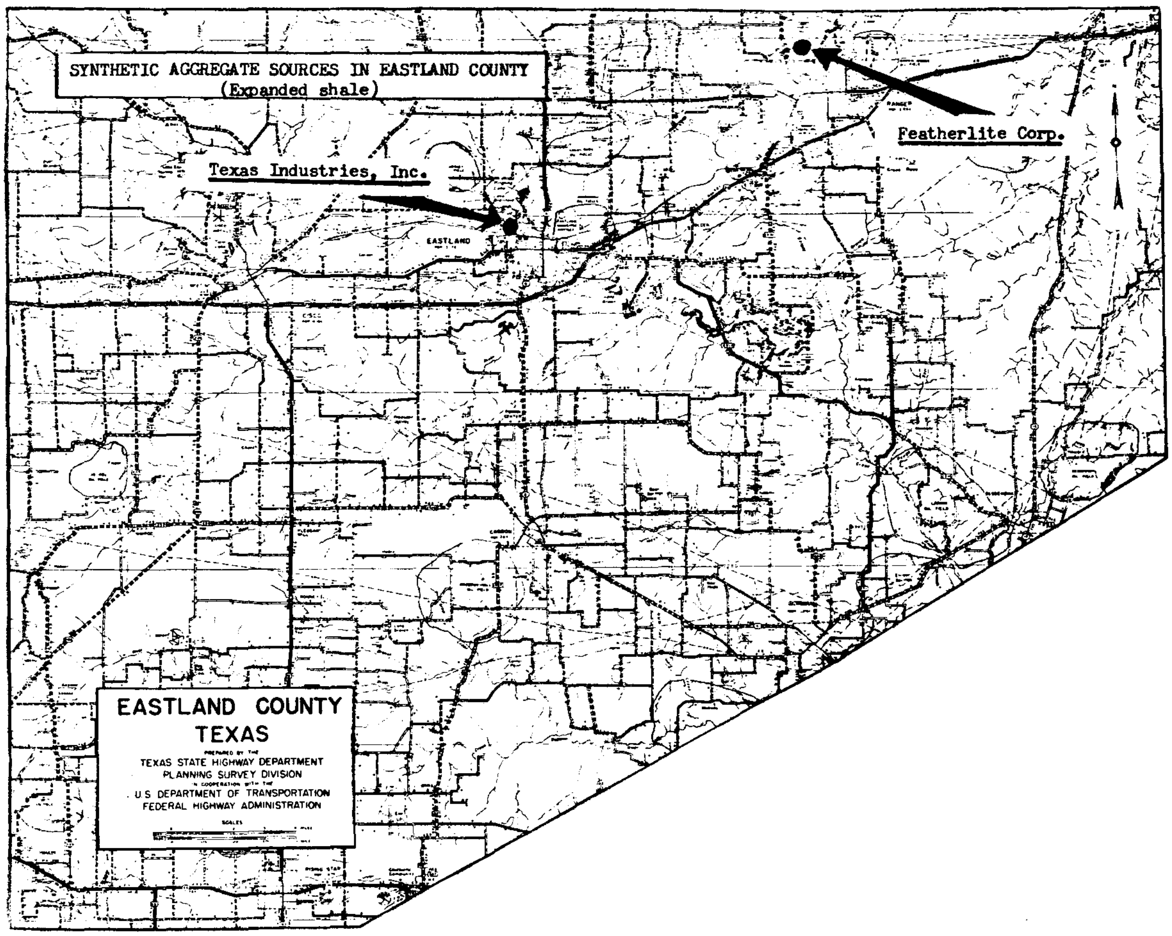
Figure 4

by the skid trailer. As can be seen, the lightweight-aggregate type increased in skid resistance and reflected a very high skid reading after several million traffic counts. This trend also correlates with observations seen with polish value determinations. The blast furnace slag and basaltic trap rock exhibited expected polishing trends in view of their polishing characteristics found by polish value tests. The data on the Wise County sources considered in this report compares well with the drop in skid-trailer readings with increasing traffic.

Data listed in Table III provides some support for the consideration of still another parameter to aid in predetermining an aggregate's susceptibility to traffic polishing. Insoluble residue analyses on carbonate aggregates suggest that the greater the insoluble residue, the better chance it has to resist polishing. Of course, there are exceptions. As can be seen in Table III, the limestone rock asphalt sample which had both relatively high skid number and polish value, had a relatively low insoluble residue content. The Wise County limestones with their low acid insoluble contents have correspondingly low polish values and skid numbers, whereas, the argillaceous limestone from near Denison, which has a low residue content, appears to have a potential for high skid resistance as indicated by polish value. However, its relatively high polish value probably can be contributed to a unique microtexture brought about by differential attrition. The sample showed evidence of rapid wear during the polish test which supports its average loss of 37% by the Los Angeles Abrasion Test (Table I). No skid trailer data is available for this source as a surface treatment aggregate because its high wear loss is recognized. The aggregate from Stringtown, with its heterogeneity as evidenced by petrographic and chemical analysis, exhibits relatively high skid resistance as measured by skid trailer and polish value tests. Acid treatment showed it to have the highest percentage of insoluble residue of all the carbonate type aggregates examined in this study. In addition, its average Los Angeles wear loss of 20% (Table I) is reflected in the material's ability to withstand years of traffic. For lack of sufficient tests, the skid trailer data on surface treatments containing the Bromide material is somewhat inconclusive. Trailer readings on flushed surfaces may reflect the fairly low reading of .33, whereas, petrographic evidence, polish value and insoluble residue indicate that a potential for skid resistance exists for the Bromide material.

APPENDIX I

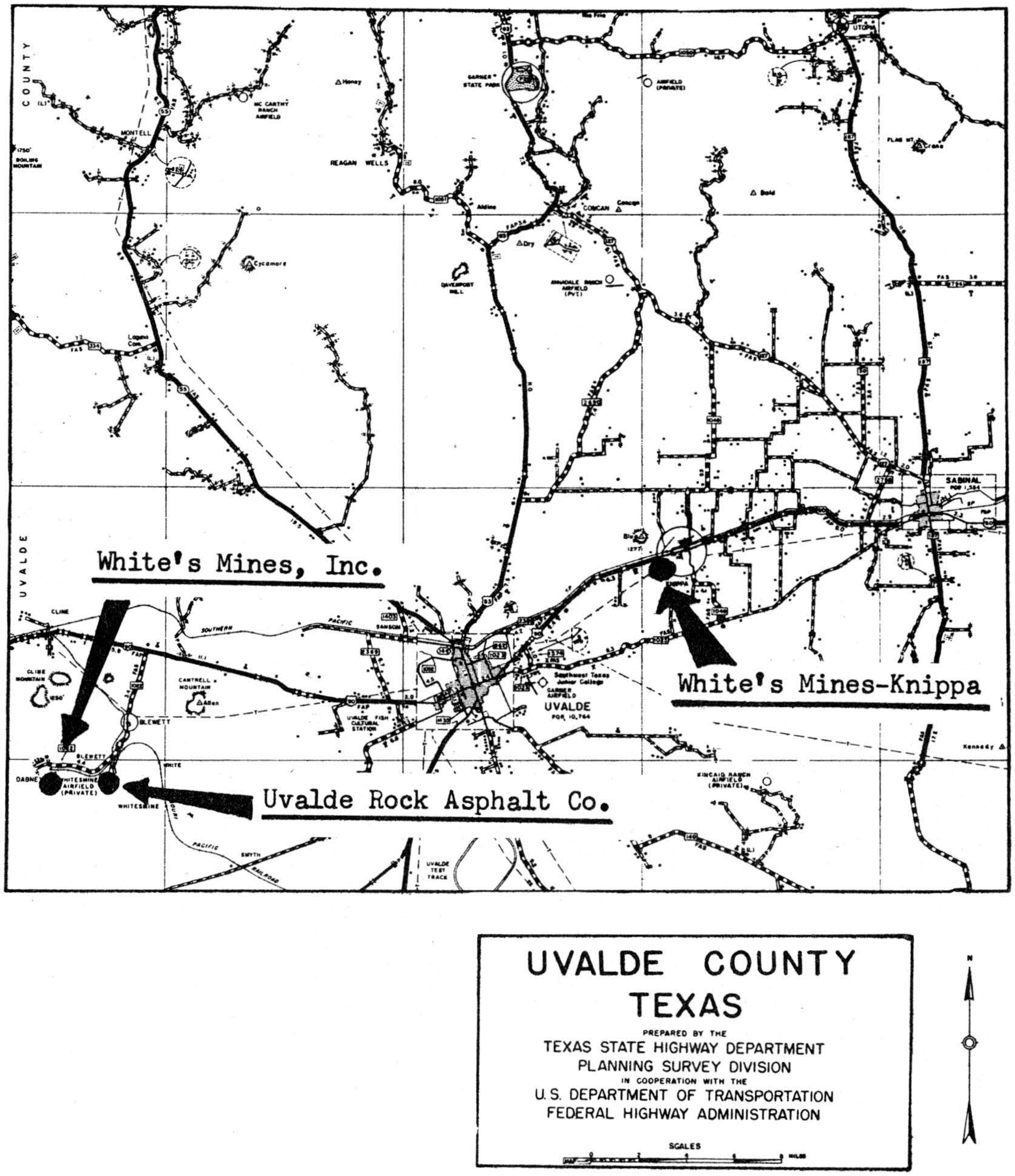

CRUSHED LIMESTONE PRODUCERS IN WISE COUNTY
Texas Industries
Vulcan
Trinity
Gifford-Hill
Lone Star
DECATUR
GENERAL HIGHWAY MAP
WISE COUNTY
TEXAS
TEXAS STATE HIGHWAY DEPARTMENT
PLANNING SURVEY DIVISION
U.S. DEPARTMENT OF TRANSPORTATION
FEDERAL HIGHWAY ADMINISTRATION



SYNTHETIC AGGREGATE SOURCES IN EASTLAND COUNTY
(Expanded shale)
Texas Industries, Inc.
Featherlite Corp.
EASTLAND
EASTLAND COUNTY
TEXAS
TEXAS STATE HIGHWAY DEPARTMENT
PLANNING SURVEY DIVISION
U.S. DEPARTMENT OF TRANSPORTATION
FEDERAL HIGHWAY ADMINISTRATION

LIMESTONE ROCK ASPHALT AND TRAP ROCK SOURCES

IN UVALDE COUNTY



APPENDIX II

APPENDIX II

PROCEDURE FOR DETERMINATION OF PER CENT BY WEIGHT OF ACID INSOLUBLE RESIUE IN CARBONATE-BEARING AGGREGATES:

1. Obtain a representative sample of production material and crushed to pass a No. 10 mesh sieve.

2. Weigh a representative 10.00 $\pm$ 0.01 gram sample into a 400 ml. beaker.

3. Place the beaker and sample on a hot plate and add slowly 300 mls. of 1:3 Hydrochloric Acid.

4. After the foaming reaction stops, add an additional 10 mls. of 1:3 Hydrochloric Acid and check to assure that the reaction has proceeded to completion.

5. Boil the mixture for 5 to 10 minutes and filter through a No. 42 Whatman filter paper.

6. Wash the residue retained on the filter paper some 3 to 4 times with hot distilled water.

7. Test the final filtrate for the presence of chloride utilizing 0.1 N Silver Nitrate solution. Repeat wash if a white silver chloride precipitate forms. Proceed to a relatively chloride free filtrate condition.

8. Transfer the filter paper and the residue to a weighted platinum crucible and ignite over a Meker type burner.

9. After the ignition over the burner, place the crucible and contents in a 2000^{o}F Muffle furnace for a period of 45 minutes.

10. Calculations: $\% \text{ Acid Insoluble Residue} = \frac{(\text{Residue wt.})\ (100)}{\text{Sample wt.}}$