

FHWA/RD-81/115
Technology for Improving the Wear Resistance
of Aggregates by Materials Impregnation
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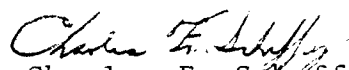
FOREWORD

This report presents the results of a study investigating the improvement of the wear-resistance properties of aggregates by impregnation. The study's objective was to evaluate various impregnation materials and techniques for their effect in improving the wear resistance of natural aggregate materials.

Five impregnants were finally selected for evaluation: three thermoplastic materials; one thermosetting material, and one inorganic material. The three thermoplastic materials were two methyl methacrylate-based systems and a styrene-based system. The thermosetting material was a water-based phenol formaldehyde resin and the inorganic material was sodium silicate. The four aggregates selected for extensive testing were: a highly porous, poorly cemented limestone; a highly porous dolomite; a fine grained, volcanic, lithic arenite (sandstone); and a highly porous, poorly cemented quartzite.

Physical testing of the impregnated and unimpregnated aggregate indicated that impregnation decreased the Los Angeles abrasion loss and the sodium sulfate soundness test loss. However, it was found that the polish resistance was not significantly improved by impregnation for any of the test aggregates. This means that an aggregate which is unsuitable for a wearing course due to its polishing characteristics could not be rendered acceptable by any of the impregnants investigated.

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Charles F. Scheffey

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16. Abstract <p>The use of impregnation as a means of improving the wear resistance of natural aggregates was investigated. A series of tests were conducted using four aggregates of varying quality, selected from a sampling of 40 different aggregates. Tests performed to evaluate the effect of impregnation on the physical, mechanical, wear resistance and polish resistance properties of the aggregates included: petrographic analysis, Los Angeles abrasion loss, sodium sulfate soundness loss, the British polish wheel, aggregate surface microtexture analysis, and two circular test track studies. The strength of concrete cast with unimpregnated and impregnated aggregate was also tested, as was the affinity of asphalt to bind to impregnated aggregate. Impregnants studied included: MMA-based and styrene-based monomer systems, water based phenol formaldehyde resin, and sodium silicate.</p> <p>In general, it was found that the physical and mechanical properties of natural aggregates can be significantly improved by impregnation. Significant improvements in wear resistance can also be obtained, however, in some instances this improvement is accompanied by a decrease in polish resistance. The use of impregnated aggregate can increase the strength of concrete. Impregnation can also reduce the tendency of some aggregates to strip.</p>			
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CONTENTS

1.0	INTRODUCTION	1
1.1	Background.	1
1.2	Objectives.	1
1.3	Approach.	2
2.0	ORGANIZATION OF RESEARCH PROGRAM	3
2.1	Literature Survey	3
2.1.1	Introduction	3
2.1.2	Properties of Wear-Resistant and Polish-Resistant Aggregates	3
2.1.3	Impregnation Technology.	4
2.2	Laboratory Test Program	5
2.3	Test Methods for Aggregate Evaluation	5
2.3.1	Petrographic Examination (ASTM C 295).	5
2.3.2	Specific Gravity and Absorption (ASTM C 127)	6
2.3.3	Los Angeles Abrasion Test (ASTM C 131)	6
2.3.4	Sodium Sulfate Soundness Test (ASTM C 88).	6
2.3.5	Accelerated British Polish Wheel Test (Tex-438-A).	6
2.3.6	Analysis of Aggregate Surface Microtexture	6
2.3.7	Circular Test Track Studies.	8
2.3.8	Static Immersion Stripping Test (ASTM D 1664).	8
2.3.9	Mechanical Properties of Concrete.	8
2.3.10	Freeze-Thaw Durability of Concrete	9
2.3.11	Concrete Mix Design.	9
2.4	Procurement of Test Aggregates.	10
3.0	PHASE ONE - TEST RESULTS	11
3.1	Selection of Aggregates for Testing	11
3.2	Selection of Impregnants.	16

4.0	PHASE TWO - TEST RESULTS	19
4.1	Introduction.	19
4.2	Evaluation of Physical and Mechanical Properties.	19
4.2.1	Los Angeles Abrasion Test Results.	19
4.2.2	Sodium Sulfate Soundness Test Results.	20
4.3	Evaluation of Wear Resistance and Polish Resistance	22
4.3.1	Accelerated Polish Wheel Tests	22
4.3.2	FHWA Microtexture Study.	27
4.3.3	Maryland Circular Test Track Study	30
4.3.4	North Carolina Circular Test Track Study	31
4.4	Evaluation of Impregnated Aggregates in Pavement Systems.	36
4.4.1	Concrete Mixes	36
4.4.2	Bituminous Mixes	40
5.0	PRELIMINARY IMPREGNATION PROCESS DESIGN AND ECONOMIC EVALUATION. . .	42
6.0	CONCLUSIONS AND RECOMMENDATIONS.	46
6.1	Conclusions	46
6.2	Recommendations	46
	REFERENCES.	47
	APPENDIX A ANNOTATED BIBLIOGRAPHY.	49
A.1	Impregnants and Impregnation Technology	49
A.2	Wear Resistance and Polish Resistance of Aggregates	54
	APPENDIX B PETROGRAPHIC ANALYSES OF TEST AGGREGATES.	64
	APPENDIX C SELECTED IMPREGNANTS AND LABORATORY IMPREGNATION PROCEDURE. .	70
C.1	Selected Impregnants.	70
C.2	Laboratory Impregnation Process	71

LIST OF TABLES

1	Summary of Petrographic Analyses12
2	Physical and Mechanical Properties of Aggregates14
3	Physical and Mechanical Properties of Aggregates Selected for Testing.15
4	Synthetic Aggregates Evaluated16
5	Properties of Aggregates Selected for Testing.17
6	Results of Los Angeles Abrasion Tests on Impregnated Aggregates19
7	Results of Sodium Sulfate Soundness Tests on Impregnated Aggregates21
8	British Polish Wheel Test Results.23
9	Microtexture Study Test Results.29
10	Polish Resistance Test Results - Maryland Circular Test Track Study.31
11	Polish Resistance Test Results - North Carolina Circular Test Track Study35
12	Strength of Concrete Cast with Unimpregnated and Impregnated Aggregate.39
13	Freeze-Thaw Test Results for Concrete Cast with Aggregate 480140
14	Static Immersion Stripping Test Results.41
15	Breakdown of Production Costs for an Impregnation Facility45
16	Material Costs of Impregnants.45

LIST OF FIGURES

1	Accelerated British Polish Wheel.	7
2	Depth Micrometer Attached to the Frame of the Polish Wheel.	7
3	Areas of Aggregate Shortages as Reported by State Highway Departments	9
4	Extended Polish Wheel Tests - Aggregate 0901.	24
5	Extended Polish Wheel Tests - Aggregate 2307.	25
6	Extended Polish Wheel Tests - Aggregate 4315.	25
7	Extended Polish Wheel Tests - Aggregate 4801.	26
8	Maryland Circular Test Track Results - Aggregate 0901	32
9	Maryland Circular Test Track Results - Aggregate 4315	33
10	Maryland Circular Test Track Results - Aggregate 4801	34
11	North Carolina Test Track Results - Aggregate 0901.	37
12	North Carolina Test Track Results - Aggregate 2307.	37
13	North Carolina Test Track Results - Aggregate 4315.	38
14	North Carolina Test Track Results - Aggregate 4801.	38
15	Production Scheme for the Manufacture of Impregnated Mineral Aggregates.	43
16	Aggregate 0901, Limestone	68
17	Aggregate 2307, Dolomite.	68
18	Aggregate 4315, Lithic Arenite.	69
19	Aggregate 4801, Quartzite	69
20	Placement of Aggregate in Impregnation Vessel	72
21	Sealed Impregnation Vessel.	72

1.0 INTRODUCTION

1.1 Background

In the interest of highway safety and energy conservation, it is essential to develop durable pavement systems that are highly wear and skid resistant. Because aggregates constitute about 75% of the volume of concrete and 80% of the volume of bituminous mixtures (1), their influence on the final properties of these materials is very significant. It is therefore important to identify sound durable aggregates with high resistance to wear and polishing, for use in paving systems. However, the supplies of natural aggregates satisfying these requirements are being rapidly depleted in many areas of the United States. As a result, large quantities of aggregates have to be transported to these regions.

Potential fuel shortages and steadily increasing transportation costs have heightened the need to examine alternatives to shipping aggregates great distances. One possible solution is to beneficiate locally available aggregate which may not satisfy all specifications. To this end, Brookhaven National Laboratory (BNL) has carried out a research program on improving the wear resistance of natural aggregates by impregnation.

Considerable research has been done at BNL since 1965 on the use of polymer impregnation to improve the strength and durability of portland cement concrete (2-6). Its results served as a basis for the work performed in this research program. They indicated, in general, that polymer impregnation of concrete produced a composite material with properties vastly superior to those of ordinary concrete, and in particular, that impregnation of extremely poor quality concrete produced a composite with properties equal to or better than those of good quality concrete. These results suggested that impregnation could be used to improve the properties of natural aggregates.

The BNL research program is part of the Federal Highway Administration's (FHWA) Federally Coordinated Program (FCP), Project 4G, "Substitute and Improved Materials to Effect Materials and Energy Conservation in Highways," an objective of which is to develop extremely durable pavement systems.

1.2 Objectives

The specific objectives of the BNL program are to investigate and evaluate materials and impregnation techniques to modify natural mineral aggregates to yield composite materials with high wear and skid resistance. Particular emphasis is on improving natural aggregates that are unacceptable for use in highway pavement systems because of their high susceptibility to wear and polishing.

1.3 Approach

Seven tasks were developed as guidelines for the research program:

- Task A. Literature Review and Procurement of Natural Aggregates.
- Task B. Impregnant Identification.
- Task C. Aggregate Characterization.
- Task D. Aggregate Impregnation and Characterization of Composite.
- Task E. Accelerated Wear/Polishing Tests.
- Task F. Impregnation Process Design and Economic Analysis.
- Task G. Documentation of Results.

2.0 ORGANIZATION OF RESEARCH PROGRAM

2.1 Literature Survey

2.1.1 Introduction

A literature survey was carried on throughout the course of the research program. Its specific objectives were (a) to determine the mechanisms involved in the wear and polishing of aggregates and to identify the properties required for high wear and polish resistance, and (b) to compile available information on potential impregnants and impregnation technology. The survey results are presented in Appendix A as an annotated bibliography.

2.1.2 Properties of Wear-Resistant and Polish-Resistant Aggregates

Selection of the impregnants to be evaluated for use in improving the resistance of natural aggregates to wear and polishing requires consideration of the mechanism of each process and of the aggregate characteristics that influence it.

Polishing is defined by Dahir and Henry (7) as the loss of aggregate microtexture by particle smoothing. Wear is defined as the loss of mineral grains or rock particles, which may result in a loss of macrotexture. It is generally agreed that wear results from stresses imposed by traffic loads and environmental changes, and polishing from abrasion of the small aggregate asperities by vehicle tires. Thus, wear and polishing involve similar processes that differ only in the degree and rate of material loss.

Although factors such as aggregate particle size, shape, and gradation have some influence on wear and polish resistance, the petrographic properties of the individual aggregate have the greatest influence. The properties cited most frequently are constituent material proportion and hardness, differential wear, and grain consolidation (7).

Mineral composition and hardness were found to be the most important. Dahir and Mullen (8) report that optimum skid resistance is obtained with an aggregate consisting of 50 to 70% of hard minerals (Mohs hardness ≥ 6 or 7) and 30 to 50% of soft minerals (Mohs hardness ≤ 2 or 3). These proportions allow for the differential wearing of the constituent minerals that is necessary for high skid resistance.

Differential wearing maintains skid resistance by continuous renewal of the aggregate surface: as the soft mineral binder is worn away, the hard mineral grains that provide the skid resistance become exposed. Before the asperities of the hard grains have worn sufficiently to become polished, the soft binder is worn down so that it is no longer able to hold the hard grains in place. The hard grains are then displaced and new unpolished grains are exposed.

The binder should be soft enough to wear at a faster rate than the individual grains, but it must be strong enough to allow a consistent and desirable rate of surface renewal. If the binder wears too quickly, the aggregate macrotexture will also wear too quickly, and this can cause adverse changes in the pavement surface texture, such as rutting.

2.1.3 Impregnation Technology

Considerable work has been done in recent years on the use of impregnation to improve the strength and durability of porous materials, notably portland cement concrete, but the concept of impregnation is by no means new. The development of resin impregnated paper for use in the electronic industry dates back to 1910. Research on the use of impregnation to stabilize wood started in the early 1940s. Studies of polymer impregnated concrete (PIC), in the United States, began in 1965 with the initiation of a joint program by Brookhaven National Laboratory (BNL) and the U.S. Bureau of Reclamation (USBR).

PIC is a composite material prepared by impregnating precast concrete with a liquid monomer which is subsequently polymerized in situ. The polymer, by filling the porous void volume of the concrete, significantly improves its strength and durability. The results of the BNL-USBR program, as well as others, gave rise to considerable worldwide interest in PIC. Potential applications for PIC include bridge deck panels, beams, piling, curbing, railroad ties, piping, and waste containment systems.

Much of the research on impregnated concrete has dealt with the use of low viscosity, liquid monomers such as methyl methacrylate (MMA) and styrene, which are subsequently converted into polymers, but a wide variety of other impregnants such as sulfur, paraffin, epoxies, polyesters, and linseed oil/mineral spirits mixtures have been investigated. The use of impregnation is based on the concept that, if the porous void volume of a material is partially or completely filled, the resulting interconnecting network of impregnant will serve as an internal reinforcing system and thus give added strength, and, by reducing the permeability of the material, will improve its durability. The basic impregnation process consists of three steps: drying the material to be impregnated, saturating it with the impregnant, and curing the impregnant in situ.

Research has shown (2,3) that, to obtain maximum loading of impregnant in a porous material such as concrete or aggregate, all the free moisture in the material must be removed. The presence of moisture within the void volume will inhibit complete penetration of the impregnant.

Methods of drying include oven drying, vacuum drying in a heated air stream, infrared heating, and microwave heating. The most convenient and most commonly used method is oven drying at a minimum temperature of 150°C (302°F). Research performed at BNL (3) indicated that concrete could be dried to a constant weight in 7 hours at 150°C (302°F). Lower temperatures required more time, and drying was not always complete. Higher drying temperatures can, in some cases, damage the material.

The impregnation process requires displacement of the air within the void volume of the material to be impregnated. This can be accomplished by several saturation techniques involving some combination of evacuation, soaking, and pressurization. The particular process used depends on the properties of the impregnant and the material to be impregnated. In the vacuum-soak-pressure technique, the material to be impregnated is first placed under vacuum to remove the air from the void volume and prevent

entrapment of air in its center. Then the impregnant is introduced into the impregnation vessel, and overpressure is applied to accelerate its penetration.

Curing depends on the type of impregnant being used. Monomers such as MMA and styrene are polymerized by a thermal-catalytic process, in which a peroxide initiator, added to the monomer prior to impregnation, decomposes, when heated, into free radicals. The free radicals initiate the polymerization reaction, converting the liquid monomer to a solid. Impregnants such as sulfur and paraffin, which are heated to get them into a liquid state, are cured simply by allowing them to cool. Water based impregnants such as sodium silicate and some phenolic resins are cured by using heat to drive off the water.

2.2 Laboratory Test Program

The laboratory test program had two phases. In Phase One, the aggregates collected from various sections of the United States were screened by determination of their physical, mechanical, wear resistance, and polish resistance properties. Based upon an evaluation of the data collected, four aggregates were selected for impregnation studies. The tests used included petrographic analysis, Los Angeles abrasion loss, determination of bulk specific gravity and water absorption, and the accelerated British polish wheel test, modified to allow measurement of the depth of wear of selected aggregate particles. Impregnants were also evaluated and selected.

In Phase Two, the effect of impregnation on the various properties of the four aggregates selected for study was evaluated, with emphasis on wear resistance and polish resistance. The tests performed included those used in Phase One and also the sodium sulfate soundness test, analysis of aggregate surface microtexture in relation to pavement frictional characteristics, polish resistance studies at the circular test tracks of the Maryland State Highway Administration and the North Carolina Department of Transportation, the static immersion stripping test for bituminous mixes, and comparison of the strength properties of concrete cast with unimpregnated and impregnated aggregate.

2.3 Test Methods for Aggregate Evaluation

The test methods used to evaluate the physical, mechanical, wear resistance, and polish resistance properties of unimpregnated and impregnated aggregates are described below.

2.3.1 Petrographic Examination (ASTM C 295)

Representative samples of each aggregate were examined microscopically in petrographic thin-sections by a commercial petrographer. The objective was to identify the mineral constituents and the physical structure of the aggregate.

2.3.2 Specific Gravity and Absorption (ASTM C 127)

The bulk specific gravity and the water absorption were determined for each of the aggregates received. The water absorption indicates how readily an aggregate can be impregnated and how much of a polymer loading can be expected. Work at BNL has shown that a polymer loading, by weight, equal to 80% of the water absorption value can easily be obtained.

2.3.3 Los Angeles Abrasion Test (ASTM C 131)

The Los Angeles abrasion test was used to evaluate the resistance of each aggregate to wear due to abrasion, crushing, and impact forces.

2.3.4 Sodium Sulfate Soundness Test (ASTM C 88)

The sodium sulfate soundness test was used to evaluate the resistance of the aggregates to simulated freeze-thaw conditions.

2.3.5 Accelerated British Polish Wheel Test (Tex-438-A)

Accelerated British polish wheel tests were done by test method Tex-438-A of the Texas State Department of Highways and Public Transportation to measure the extent to which coarse aggregate will polish. This test is the same as outlined by ASTM D 3319-74T, except that the duration of the test is reduced from 10 to 9 hours, the silicon carbide grit is fed at a rate of 6 ± 2 g/min rather than 25 ± 5 g/min, and the water is fed at a rate of 50 to 75 ml/min.

The degree of polishing was determined using the British Portable Tester (ASTM E 303) and expressed as the polish value (PV) of the aggregate. The PV's reported are those read directly from the scale used to measure the British Portable Numbers, not those measured with the smaller (and harder to read) auxiliary scale originally intended for use with the polish wheel.

To measure the depth of wear of the aggregates caused by the polishing action, a depth micrometer was mounted to the frame of the British polish wheel. Depth measurements were taken for selected aggregate particles before, during, and after the polish test (Figures 1 and 2).

Polish value requirements for aggregates to be used in the construction of highway pavements in Texas are based on traffic density. Roadways carrying < 750 vehicles per day require no minimum PV, others may require a minimum PV of 30 to 33, and those used by > 5000 vehicles per day require a $PV \geq 35$.

2.3.6 Analysis of Aggregate Surface Microtexture

In a study done with the assistance of the Federal Highway Administration, Office of Research, Materials Division, to evaluate aggregate surface microtexture in relation to aggregate frictional characteristics, the surface microtexture of selected aggregate particles in British polish wheel test coupons was measured with the FHWA automated image

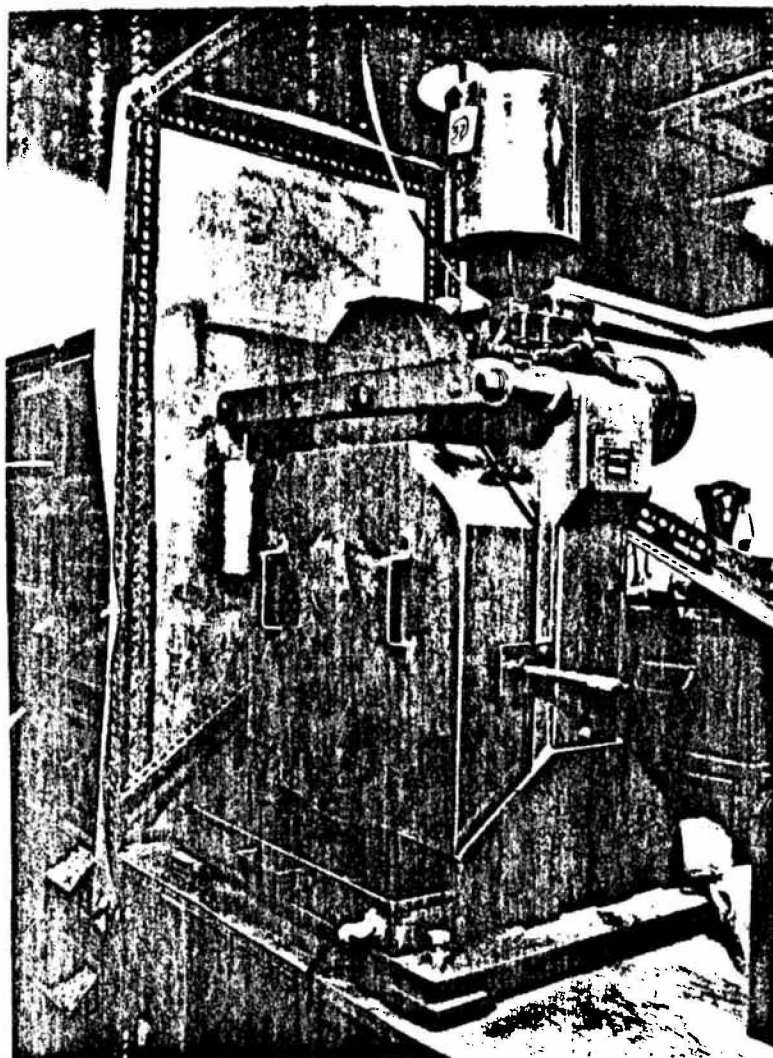


Figure 1 Accelerated British Polish Wheel.

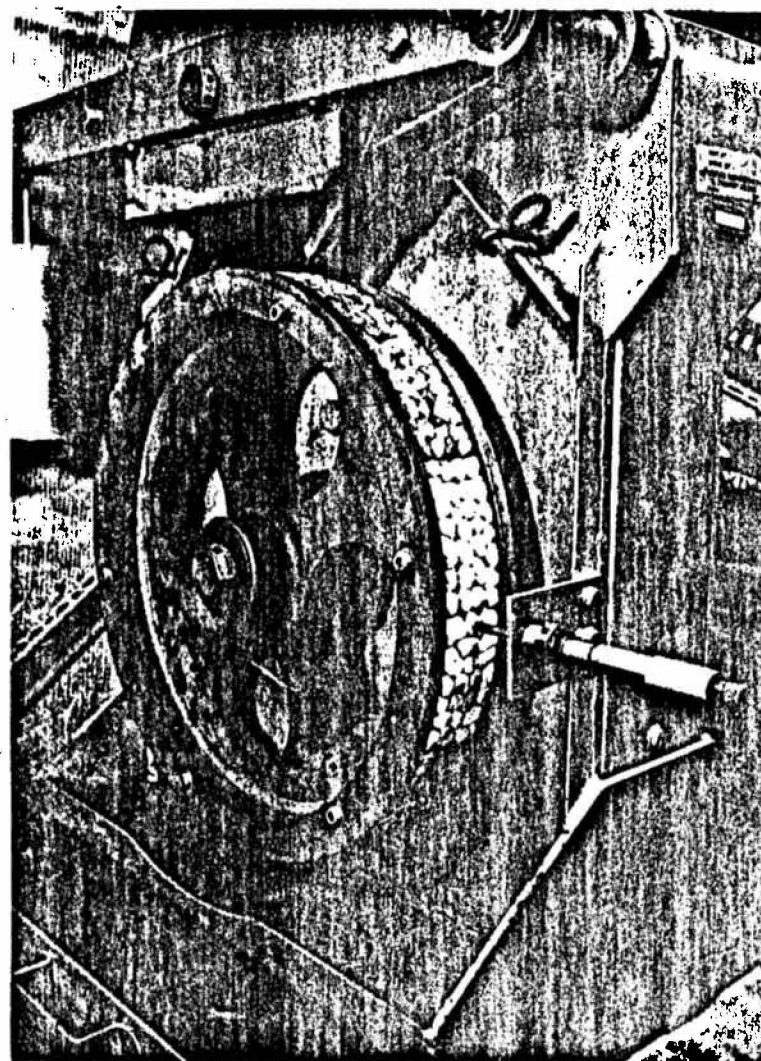


Figure 2 Depth Micrometer Attached to the Frame of the Polish Wheel.

analysis system (9) before and after the coupons were run on the polish wheel. The data were compared to determine whether impregnation had any adverse effects on the microtexture characteristics of the aggregates studied.

2.3.7 Circular Test Track Studies

Studies were done at two laboratory scaled circular test tracks, those of the Maryland State Highway Administration and the North Carolina Department of Transportation, to evaluate the polishability of unimpregnated and impregnated aggregate under simulated traffic conditions.

The Maryland test track (10) consists of a 1.83-m (6-ft) diameter track around which 16 trapizodal specimens are placed. Two standard size pneumatic tired automobile wheels are used to polish the test specimens, which are prepared so as to produce a plane surface consisting of exposed particles of coarse aggregate held in place by cement mortar. Aggregate polishability is evaluated by measuring the frictional resistance of the specimen surface with the British Portable Tester (ASTM E 303) and by determining the polish strain value. The polish strain value is calculated from strain measurements recorded by gauges mounted on the arm controlling the movement of a spinning third wheel which is passed over the track at a peripheral speed of 30 mph (48 kmph). The gauges measure the resistance of the test specimen surface to the slippage of the test tire.

The North Carolina test track (11) consists of a 0.91-m (3-ft) diameter track around which are placed 12 test specimens made from an open-graded friction course bituminous mix into which the aggregates to be tested have been incorporated. The specimens are polished with four smooth tread go-kart tires. The skid resistance is evaluated with the British Portable Tester (ASTM E 303) and the Variable Speed Friction Tester (VST) (ASTM E 707).

The VST is a dynamic pendulum tester similar to the British Portable Tester, used to measure energy loss when a smooth tread pneumatic tire is passed over the test surface; the loss is reported as the Variable Speed Test Number (VSN). The test speed is varied by changing the spray velocity of a sheet of water directed across the surface of the specimen during testing to obtain VSN values for different test velocities.

2.3.8 Static Immersion Stripping Test (ASTM D 1664)

The static immersion stripping test was used to evaluate the affinity of AC-20 asphalt to bind to unimpregnated and impregnated aggregates.

2.3.9 Mechanical Properties of Concrete

The effect of the use of impregnated aggregate on the mechanical properties of concrete was evaluated by means of compressive, tensile splitting, and flexure tests (ASTM C 39, C 496, and C 78, respectively).

2.3.10 Freeze-Thaw Durability of Concrete

Freeze-thaw tests were done on a limited number of cylinders in accordance with the cycling procedure specified in ASTM Resistance of Concrete to Rapid Freezing and Thawing (C 666-76), Procedure A. Freeze-thaw durability was evaluated by determining the compressive strength at 0, 25, and 50 cycles.

2.3.11 Concrete Mix Design

All concrete test cylinders and beams were cast with a mix having a cement factor of 7.92 sacks/m³ (6.05 sacks/yd³), a water-cement ratio of 0.45, an air content of 5±1%, and a design slump of 7.6 to 10.2 cm (3 to 4 in.). The fine aggregate was a graded silica sand with a fineness modulus of 2.42. The coarse aggregate was soaked in tap water for 24 hr before incorporation into the concrete mix. All concrete test specimens were cured in saturated lime water for 28 days before being tested.

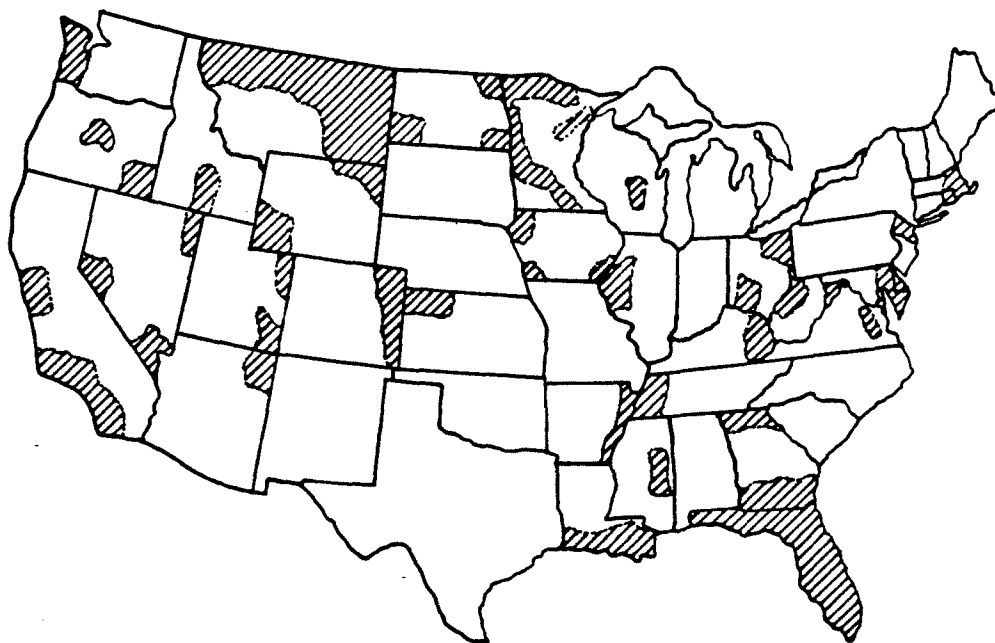


Figure 3 Areas of Aggregate Shortages as Reported by State Highway Departments (12).

2.4 Procurement of Test Aggregates

The supply of natural aggregate in the United States is so vast as to be inexhaustible, but its geographic distribution is such that the amount of available high-quality aggregate is insufficient to meet the needs of many regions of the country (see Figure 3). BNL asked thirteen state highway departments to supply samples of locally available aggregates, both those currently being used for highway pavement construction and those unacceptable for use because of poor wear and/or polish resistance. Eight of these departments were in states reporting shortages. Aggregates from 40 different sources were received from eleven states (Florida, Georgia, Indiana, Louisiana, Massachusetts, Minnesota, New York, Oklahoma, Texas, Washington and West Virginia).

3.0 PHASE ONE - TEST RESULTS

3.1 Selection of Aggregates for Testing

Of the aggregates from 40 different sources in eleven states, 24 were selected for evaluation in Phase One. Initial evaluation was based on the results of a series of five tests: petrographic analysis, Los Angeles abrasion loss, bulk specific gravity, water absorption, and the British Polish Wheel. The results are summarized in Tables 1 and 2.

After evaluation of the test results, two carbonate and two siliceous aggregates were selected for use in Phase Two of the program. One aggregate of each type was marginal and the other of good quality so that the effects of impregnation upon both poor and good quality aggregates could be tested. The carbonate aggregates selected for evaluation were Aggregate 0901 and Aggregate 2307. The siliceous aggregates selected for evaluation were Aggregate 4801 and Aggregate 4315. The characteristics of each that caused it to be chosen are discussed below.

Aggregate 0901 - This limestone was selected because of its low wear resistance. Of the 24 aggregates examined, this one showed the greatest wear in the polish wheel test and the second greatest wear due to abrasion. This is not surprising in view of the petrographic analysis: the aggregate consists almost entirely of pure, fine grained calcite, which has a Mohs hardness of 3, with only a small amount of cement between the grain particles, and about 20% pore space by volume. These characteristics suggest a soft aggregate with low wear and abrasion resistance, and easy to impregnate because of the high porosity. This aggregate has a good polish value (PV) even though it is mostly calcite. This is probably because of the vesicular nature of the aggregate. Therefore, impregnation, which partially or completely fills the pore volume, may reduce its polish resistance. The above information suggests requirements regarding the impregnant chosen for improvement of this aggregate. (a) The impregnant must help reinforce the aggregate by cementing the grain particles together. (b) To prevent reduction of the polish resistance, the impregnant, after curing, should be significantly harder than the calcite. In this case the impregnant will serve as the hard phase of the aggregate and the calcite as the soft phase.

Aggregate 2307 - This dolomite was selected because of its similarity to Aggregate 0901 except for wear resistance. Both are relatively homogeneous, highly porous aggregate with marginal cementing of the grain particles, but these characteristics do not seem to lower the wear resistance of Aggregate 2307. Its wear resistance, as measured by the Los Angeles abrasion test is good; as measured on the British Polish Wheel, it is average when compared with that of the other aggregates tested. This aggregate also has a good polish value (PV). The objective of impregnation is to improve the wear resistance by reinforcing the cementing of the grain particles.

Aggregate 4801 - This quartzite was selected because of its extremely poor resistance to wear due to abrasion. It had the highest Los Angeles Abrasion loss (79%) of all the aggregates tested. However, it has good wear resistance as measured on the British Polish Wheel, and very good polish resistance. The petrographic analysis shows that this quartzite, like the

Table 1 Summary of Petrographic Analyses

Aggregate Code No.	Rock Type	General Petrographic Description
0901	Limestone	A highly porous, poorly cemented limestone consisting of nearly pure, fine grained calcite.
1001	Limestone	A uniform grained limestone consisting of nearly pure calcite and small amounts of quartz.
1008	Limestone	A limestone consisting of nearly pure calcite with small amounts of quartz.
1009	Granite Gneiss	A rock consisting of an intertwinning composite of quartz, plagioclase, and microcline.
1401	Dolomitic Limestone	A fine grained, homogeneous dolomitic limestone characterized by random fracturing of the rock.
1403	Dolomite	A uniform grained, pure dolomite characterized by an uneven distribution of porous and non-porous regions which suggests a high variability in mechanical properties.
2101	Silica Quartzite	A texturally heterogeneous rock consisting primarily of fine grained quartz and coarse grained feldspar.
2104	Quartz- Muscovite Schist	A compositionally and texturally heterogeneous rock consisting primarily of quartz, chlorite, and muscovite.
2303	Basalt	A highly altered, porphyritic basalt consisting of augite set in a fine grained chlorite matrix.
307	Dolomite	A highly porous, poorly cemented, homogeneous dolomite.
3201	Quartz Arenite	A moderately sorted, very fine grained, sub-mature quartz arenite.
3603	Limestone	Texturally, a moderately homogeneous limestone consisting of nearly pure calcite with a small amount of quartz.

Table 1 Summary of Petrographic Analyses (Continued)

Aggregate Code No.	Rock Type	General Petrographic Description
3604	Limestone	A very pure limestone consisting of a matrix of submicroscopic calcite, which in localized areas is recrystallized with an increase in grain size.
3606	Lithic Arenite (Sandstone)	A moderately porous sandstone consisting primarily of quartz grains weakly cemented together along straight, unsutured contacts.
3607	Orthoquartzite	The rock consists of detrital quartz and feldspar with calcite cement.
4305	Limestone	A highly porous limestone consisting of fine grained calcite and small amounts of quartz.
4306	Limestone	A very soft, extremely pure, fine grained limestone.
4307	Limestone	A heterogeneously grained limestone consisting of nearly pure calcite with small amounts of quartz.
4313	Subarkose	A well sorted, fine grained, mature subarkose consisting of detrital fragments of quartz, chert, and feldspar loosely cemented by secondary quartz and iron oxide.
4315	Lithic Arenite (Sandstone)	A predominantly fine grained, volcanic lithic arenite consisting of detrital fragments of quartz, plagioclase, and alkali feldspars.
4702	Monzonite	The rock consists of a closely interlocking fabric of fine grained minerals, most of which are plagioclase and alkali feldspars.
4703	Syenite	A fine grained rock consisting primarily of alkali feldspar and perthite.
4704	Basalt	The rock consists mainly of an inhomogeneous arphanitic groundmass with small amounts of augite and magnetite(?) occurring as disseminated phenocrysts.
4801	Quartzite	The rock is a moderately well sorted, porous quartzite consisting primarily of detrital grains of quartz with fragments of feldspars, muscovite, biotite, and chlorite.

Table 2 Physical and Mechanical Properties of Aggregates

Aggregate Code No.	Rock Type	Bulk Specific Gravity	Water Absorption, Wt%	L.A. Abrasion Loss, Wt%	British Polish Wheel ^a	
					PV	Wear, mm
0901	Limestone	2.28	4.32	42	37	1.1988
1001	Limestone	2.69	0.56	20	28	0.4064
1008	Limestone	2.68	0.48	19	34	0.4521
1009	Granite Gneiss	2.61	0.51	—	35	0.4445
1401	Dolomitic Limestone	2.62	1.63	—	30	0.6350
1403	Dolomite	2.71	0.80	—	29	0.5842
2101	Silica Quartzite	2.70	0.76	23	—	—
2104	Quartz-Muscovite Schist	2.78	0.52	21	—	—
2303	Basalt	2.93	0.52	—	—	—
2307	Dolomite	2.58	3.11	28	37	0.5055
3201	Quartz-Arenite	2.57	2.60	19	48	0.3556
3603	Limestone	2.66	0.66	—	28	0.3302
3604	Limestone	2.71	0.40	25	28	0.2963
3606	Lithic Arenite	2.43	3.84	28	49	0.5080
3607	Orthoquartzite	2.65	1.04	24	40	0.3090
4305	Limestone	2.47	3.45	31	35	0.8128
4306	Limestone	2.49	2.81	28	32	0.9398
4307	Limestone	2.61	1.52	25	33	—
4313	Subarkose	2.33	3.13	26	41	0.3048
4315	Lithic Arenite	2.10	5.58	22	36	0.3454
4702	Monzonite	2.76	1.60	—	—	—
4703	Syenite	2.78	1.24	—	33	0.4572
4704	Basalt	2.88	0.37	14	35	0.4725
4801	Quartzite	2.46	3.50	79	43	0.3886

^apv = polish value after 9 hours; Wear = depth of wear (mm), measured at the end of 9 hr.

carbonate aggregates chosen, is characterized by poor cementing of the individual grains and a large pore volume, which undoubtedly accounts for its poor abrasion resistance. It has good polish resistance because it consists largely of quartz, which has a Mohs hardness of 7. The objective of impregnation is to reinforce the cementing of the grains without adversely affecting the polish resistance.

Aggregate 4315 - This lithic arenite (sandstone) was selected primarily to determine whether an aggregate that already has good wear resistance and polish resistance can be improved by impregnation.

The physical and mechanical properties of the selected aggregates are summarized in Table 3. Detailed petrographic descriptions of the four chosen aggregates are given in Appendix B.

British polish wheel tests were also done on five synthetic aggregates to compare their wear resistance and polish resistance with those of natural aggregates. The results were used to select a control aggregate for subsequent polish wheel tests, to be used as a reference in comparing polish wheel data from different tests. They are given in Table 4, with descriptions of the synthetic aggregates tested. Of the five synthetic aggregates tested, the expanded clay had the greatest polish resistance, but it also had the least resistance to wear. In contrast, the Mulcoa-70 calcined clay had the lowest polish resistance and the best wear resistance. The calcined Guyana bauxite exhibited the best overall wear resistance and polish resistance and was therefore selected for use as the control aggregate for the polish wheel tests. Calcined Guyana bauxite is generally considered to be one of the best wear resistant and polish resistant synthetic aggregates available. The polish value of 40 measured at BNL is very close to the values of 42 to 46 reported by James (13).

Table 3. Physical and Mechanical Properties of Aggregates Selected for Testing

Property	Aggregate Code No.			
	0901	2307	4315	4801
Rock Type	Limestone	Dolomite	Lithic Arenite	Quartzite
Bulk Specific Gravity	2.28	2.58	2.10	2.46
Water Absorption, wt%	4.32	3.11	5.58	3.50
L.A. Abrasion Loss, wt%	42	28	22	79
Sodium Sulfate Soundness Loss, wt%	15.0	12.8	7.8	79.2
Polish Wheel Data				
Polish Value (PV)	37	37	36	43
Depth of Wear, mm	1.1988	0.5055	0.3454	0.3886

Table 4 Synthetic Aggregates Evaluated

Aggregate Code No.	Aggregate Type (Trade Name)	Constituent Minerals, Typical Percentages	Specific Gravity	Polish Wheel Test ^a	
				PV	Wear, mm
1803	Expanded Clay	Manufactured from clay comprised predominately of the Smectite group of clay minerals and Illite.	—	49	0.4792
5101	Calcined Guyana Bauxite	Al ₂ O ₃ - 88.0 Si O ₂ - 6.5 Fe ₂ O ₃ - 3.3 Ti O ₂ - 2.0	3.10	40	0.2642
5102	Calcined Clay (Ione "C")	Al ₂ O ₃ - 41.8 Si O ₂ - 54.3 Fe ₂ O ₃ - 0.8 Ti O ₂ - 2.6	2.56	27	0.3696
5103	Calcined Clay (Mulcoa-47)	Al ₂ O ₃ - 47.8 Si O ₂ - 49.3 Fe O ₃ - 1.8 Ti O ₂ - 1.0	2.64	29	0.2307
5104	Calcined Clay (Mulcoa-70)	Al ₂ O ₃ - 70.5 Si O ₂ - 25.3 Fe ₂ O ₃ - 2.7 Ti O ₂ - 1.4	2.85	23	0.2078

^aBritish accelerated polish wheel test; PV = polish value at 9 hr; Wear = depth of wear after 9 hr.

3.2 Selection of Impregnants

A number of interrelated factors need to be considered in the selection of an impregnant for a particular application. The properties of the raw impregnant such as viscosity, vapor pressure, and curing requirements, must be considered in designing and developing the impregnation process, as well as its cost and availability and any special safety requirements in its handling and storage. The properties of the cured impregnant, such as its mechanical properties, corrosion resistance, and thermal stability must also be considered to ensure compatibility with the environment in which the composite is to be used. The factors considered in choosing the impregnants to be evaluated are discussed below.

Raw Impregnants - (a) The impregnant should have a relatively low viscosity to ensure complete penetration of the aggregate. Use of a low viscosity impregnant reduces the time required for full impregnation, helps

eliminate the need for high-pressure equipment, and lessens the likelihood of formation of a coating on the aggregate which can detrimentally effect skid resistance. (b) Curing should be as quick and as easy as possible, without the need for elaborate equipment. (c) The impregnant should be readily available and as inexpensive as possible in order to keep the entire process economically feasible.

Cured Impregnant - (a) The cured impregnant should be compatible with both concrete and bituminous mixes. (b) It should not lower the wear resistance and skid resistance of the aggregates that are impregnated. (c) It must be thermally stable at 135° to 150°C (275° to 302°F), the normal temperature range for mixing asphalt and aggregates, if the impregnated aggregate is to be used in bituminous mixes. (d) Its Mohs hardness should be sufficiently different from that of the major constituents of the aggregate to allow for differential wearing.

A number of impregnants were considered for evaluation, including methyl methacrylate (MMA)-based systems, styrene-based systems, epoxies, sulfur, paraffin, polyesters, water-based phenol formaldehyde, and sodium silicate (water glass). Five were selected; the first three were thermoplastic materials, the fourth a thermosetting material, and the fifth an inorganic material. These are listed, with some of their properties in Table 5.

Table 5 Properties of Impregnants Selected for Testing

Impregnant ^a	Viscosity (Monomer) mPa·s	Specific Gravity (Monomer)	Thermal Stability, °C			Mohs Hardness (Polymer)	Density (Polymer) g/cm ³
			Percent Weight Loss				
			0.5	1.0	5.0		
1. 90 wt% MMA - 10 wt% TMPTMA	1	0.941	140	170	230	4	1.191
2. 83 wt% MMA - 5 wt% TMPTMA - 12 wt% PMMA	15	0.965	115	140	210	4	1.185
3. 57 wt% Sty - 38 wt% TMPTMA - 5 wt% PSty	15	0.962	200	250	350	4	1.000
4. Water-Based Phenol Formal- dehyde Resin	190 ^c	—	200	235	430	4	0.992
5. Sodium Silicate	257 ^c	1.400	150	175	285	—	—

^aMMA = methyl methacrylate; TMPTMA = trimethylolpropane trimethacrylate;

PMMA = poly(methyl methacrylate); Sty = styrene; Psty = polystyrene.

^bviscosity at 24°C; 1 mPa·s = 1 cP.

^cViscosity can be adjusted by adding water.

In general, the two MMA-based systems, the styrene-based system, and the phenol formaldehyde resin were used with each of the four test aggregates. The sodium silicate was selected specifically for use with Aggregate 0901, the very soft, pure limestone, the idea being that impregnation with a material consisting largely of silica, which has a Mohs hardness greater than that of calcite, would improve the skid resistance of this aggregate. All of the impregnants except the MMA-TMPTMA-PMMA should have sufficient thermal stability to be used with bituminous mixtures.

A complete description of each impregnant and of the laboratory impregnation procedure is presented in Appendix C.

4.0 PHASE TWO - TEST RESULTS

4.1 Introduction

This chapter presents the results of tests performed to evaluate the effect of impregnation on the physical, mechanical, wear resistance, and polish resistance properties of the four selected aggregates.

4.2 Evaluation of Physical and Mechanical Properties

Four characteristics were routinely measured to evaluate the physical and mechanical properties of each batch of impregnated aggregate: bulk specific gravity, water absorption, Los Angeles abrasion loss, and sodium sulfate soundness loss.

4.2.1 Los Angeles Abrasion Test Results

The Los Angeles abrasion test was performed to evaluate the ability of the unimpregnated and impregnated aggregates to resist wear due to abrasion, crushing, and impact forces. The results are summarized in Table 6.

Table 6 Results of Los Angeles Abrasion Tests of Impregnated Aggregates

Aggregate Code No.	L.A. Abrasion Loss, wt% (Unimpregnated)	Impregnant	Impregnant Loading, wt%	L.A. Abrasion Loss wt% (Impregnated)
0901	42	MMA-TMPTMA ^a	3.44	34
		MMA-TMPTMA-PMMA ^a	4.71	21
		Sty-TMPTMA-PSty ^a	6.95	20
		Sodium Silicate 100 ^b	2.33	30
		Sodium Silicate 84 ^c	1.88	33
		Phenol-Formaldehyde ^d	11.47	16
2307	28	MMA-TMPTMA	2.42	16
		MMA-TMPTMA-PMMA	2.19	16
		Sty-TMPTMA-PSty	2.92	16
4315	22	MMA-TMPTMA-PMMA	3.12	16
		Sty-TMPTMA-PSty	5.40	15
4801	79	MMA-TMPTMA	2.15	46
		MMA-TMPTMA-PMMA ^e	1.78	35
		MMA-TMPTMA-PMMA	1.99	18
		Sty-TMPTMA-PSty ^e	1.91	33
		Sty-TMPTMA-PSty	2.27	20
		Phenol-Formaldehyde	5.19	18

^aDefined in Table 5.

^b100 wt% sodium silicate solution, viscosity = 257 mPa's. ^c84 wt% sodium silicate solution - 16% water, viscosity = 23 mPa's.

^d100 wt% phenol-formaldehyde resin. Aggregates had heavy coating of cured resin.

^eMonomer system contains no silane coupling agent.

All of the impregnants produced reductions in the abrasion loss of each of the aggregates. The most significant improvements were noted in Aggregates 0901 and 4801, which are very poorly cemented and highly porous, and had the least abrasion resistance of the four aggregates studied.

The reduction in abrasion loss for Aggregate 0901 varied between 19 and 62%. The phenol formaldehyde resin produced the greatest improvement in abrasion resistance, but it left a heavy surface coating on the aggregate which could adversely affect the polish resistance. Impregnation with the MMA-TMPTMA system provided the least improvement in abrasion resistance. The MMA-TMPTMA-PMMA system and the Sty-TMPTMA-PSty system produced the most improvements in abrasion resistance without leaving a noticeable coating of impregnant on the aggregate.

The reduction in abrasion loss for Aggregate 4801 varied between 44 and 77%. The phenol formaldehyde resin and the MMA-TMPTMA-PMMA system, with the silane coupling agent, produced the greatest improvement in abrasion resistance. However, the phenol formaldehyde resin left a heavy coating of resin on the surface of the aggregate. The MMA-TMPTMA-PMMA and the Sty-TMPTMA-PSty systems, without the silane coupling agent, both produced improvements in abrasion resistance. However, the improvements were not as great as those obtained using the silane coupling agent. The aggregate impregnated with the MMA-TMPTMA system showed the least improvement in abrasion resistance.

The three impregnants used to impregnate Aggregate 2307 (MMA-TMPTMA, MMA-TMPTMA-PMMA, and Sty-TMPTMA-PSty) all reduced the abrasion loss by 43%.

The two impregnants used to impregnate Aggregate 4315 (MMA-TMPTMA-PMMA and Sty-TMPTMA-PSty) reduced the abrasion loss by 27% and 32%, respectively.

The improvements in the abrasion resistance of the aggregates are due principally to the internal reinforcement provided by the interconnecting network of impregnant developed throughout the pore volume.

4.2.2 Sodium Sulfate Soundness Test Results

Sodium sulfate soundness tests were performed to evaluate the resistance of unimpregnated and impregnated aggregates to simulated weathering. The results are summarized in Table 7. Significant reductions in sulfate soundness loss were noted for all the aggregates as the results of impregnation. The greatest reductions in sulfate soundness loss were noted for Aggregate 4801, for which the loss dropped from 79.2 wt% to 2.8 wt% with the MMA-TMPTMA-PMMA system and to 3.9 wt% with the Sty-TMPTMA-PSty system. Some aggregates, such as Aggregates 2307 and 4315, showed an apparent relationship between impregnant loading, water absorption, and soundness loss: the greater the impregnant loading, the greater the reduction in the water absorption and in the soundness loss.

Table 7 Results of Sodium Sulfate Soundness Tests on Impregnated Aggregates

Aggregate Code No.	Control Values			Impregnant ^b	Impregnated Values			
	Bulk Specific Gravity	Water Absorption, wt%	Soundness Loss, wt% ^a		Impregnant Loading, wt%	Bulk Specific Gravity	Water Absorption, wt%	Soundness Loss, wt%
0901	2.28	4.32	15.0	MMA-TMPTMA-PMMA	4.71	2.29	1.99	0.5
				Sty-TMPTMA-PSty	6.95	2.36	0.76	0.6
				Sodium Silicate 100	2.33	2.34	2.44	0.7
				Sodium Silicate 84	1.88	2.31	2.65	0.2
2307	2.58	3.11	12.8	MMA-TMPTMA-PMMA	2.18	2.57	1.04	1.0
				Sty-TMPTMA-PSty	3.13	2.57	0.50	0.2
4315	2.10	5.58	7.8	MMA-TMPTMA-PMMA	3.12	2.16	2.34	1.9
				Sty-TMPTMA-PSty	5.40	2.16	2.19	0.9
4801	2.46	3.50	79.2	MMA-TMPTMA-PMMA	2.04	2.48	1.12	2.8
				Sty-TMPTMA-PSty	2.30	2.48	1.36	3.9

^aSodium sulfate soundness loss; allowable limit = 12 wt% loss.

^bDefined in Table 4.1

4.3 Evaluation of Wear Resistance and Polish Resistance

Four tests were used to evaluate the effectiveness of impregnation in improving the wear resistance of natural aggregates: the British polish wheel test, a circular test track study at the Maryland State Highway Administration test track in Brooklandville, Maryland, a circular test track study at the North Carolina Department of Transportation test track in Raleigh, North Carolina, and microtexture analysis of British polish wheel test coupons. The results are discussed below.

4.3.1 Accelerated Polish Wheel Tests

Tests were done with the British Accelerated Polishing Machine both to evaluate the effectiveness of impregnation as a means of improving the wear resistance of natural aggregates and to determine its influence on polish resistance. The latter was done by comparing the polish values (PV) of the unimpregnated and impregnated aggregates after 9 hr on the polish wheel. The PV of each aggregate was measured before it was run on the polish wheel to determine whether a coating of impregnant was left on the surface of the aggregate. A significant coating could adversely effect the bond between the aggregate and the matrix of a concrete or bituminous mix, as well as influencing the aggregate's polish resistance.

Wear resistance was evaluated by measuring the amount of wear exhibited by selected aggregates in the polish wheel coupons at intervals throughout the test. The measurements were made with a depth micrometer mounted on the frame of the polish wheel. To evaluate the rate of wear over an extended period, the aggregates were tested on the polish wheel for a total of 18 hr. The rate of wear was determined by a linear regression analysis on the depth of wear data collected for each aggregate. The results are summarized in Table 8 and Figures 4 through 7. Data on the wear resistance and polish resistance of calcined Guyana bauxite (Aggregate 5101) are given for comparison.

With one exception, all the aggregates exhibited reductions in wear as a result of being impregnated. No improvements were noted in polish resistance due to impregnation; in fact, about half of the impregnated aggregates showed significant reductions, that is, a PV reduction of more than 2 units (± 2 units is the normal range of experimental variation). All but one of these were carbonate aggregates. The test results for each aggregate are presented below.

Aggregate 0901 - Each of the five impregnants evaluated improved the wear resistance of Aggregate 0901 but also reduced the polish resistance significantly. The reductions in the initial PV's indicate that a coating was left on the surface of the aggregate by each impregnant. The reductions in the 9 hr PV's were probably caused by a lower than anticipated wear differential between the constituent minerals of the aggregate and the cured impregnants. In addition, filling of the pore volume changed the vesicular nature of the aggregate and thus reduced the polish resistance.

Table 8 British Polish Wheel Test Results

Aggregate Code No.	Impregnant ^b	Impregnant Loading, wt%	Polish Value (PV)		Depth of Wear, mm		Rate of Wear 10 ⁻³ mm/hr
			Initial ^d	At 9 hr ^c	At 9 hr	At 18 hr	
0901	Control	—	50	37	1.1989	2.2555	116.8
	MMA-TMPTMA	4.18	47	34	1.2090	1.9888	101.6
	MMA-TMPTMA-PMMA	5.80	46	32	0.8433	1.1532	55.9
	Sty-TMPTMA-PSty	6.95	46	31	0.7493	1.2573	63.5
	Sodium Silicate 100	2.33	40	29	0.6756	1.3386	78.7
	Sodium Silicate 84	1.88	42	31	0.7925	1.5037	78.7
2307	Control	—	47	36	0.5055	0.8306	33.0
	MMA-TMPTMA	2.71	50	36	0.4826	0.7671	30.5
	MMA-TMPTMA-PMMA	2.37	38	29	0.3886	0.5613	25.4
	Sty-TMPTMA-PSty	3.13	43	30	0.3404	0.5410	25.4
4315	Control	—	43	36	0.3454	0.6198	27.9
	MMA-TMPTMA-PMMA	4.19	43	35	0.2515	0.4775	20.3
	Sty-TMPTMA-PSty	5.40	43	35	0.2540	0.4420	17.8
4801	Control	—	49	43	0.3886	0.5156	15.2
	MMA-TMPTMA	1.82	51	44	0.3912	0.6375	20.3
	MMA-TMPTMA-PMMA	1.29	48	41	0.2743	0.4496	17.8
	Sty-TMPTMA-PSty	2.90	47	39	0.2413	0.4420	20.3
5101	—	—	50	40	0.2642	0.3988	17.8

^aDefined in Table 6.^bPolish value of coupon before being tested on polish wheel.^cPolish value of coupon after being tested on polish wheel for 9 hr.

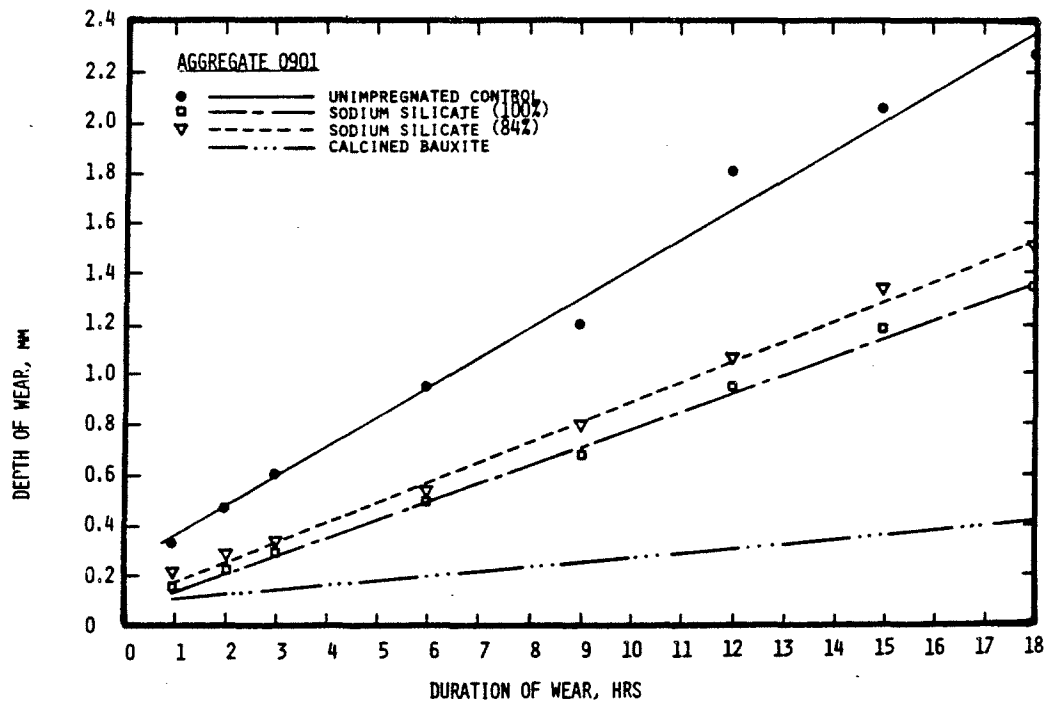
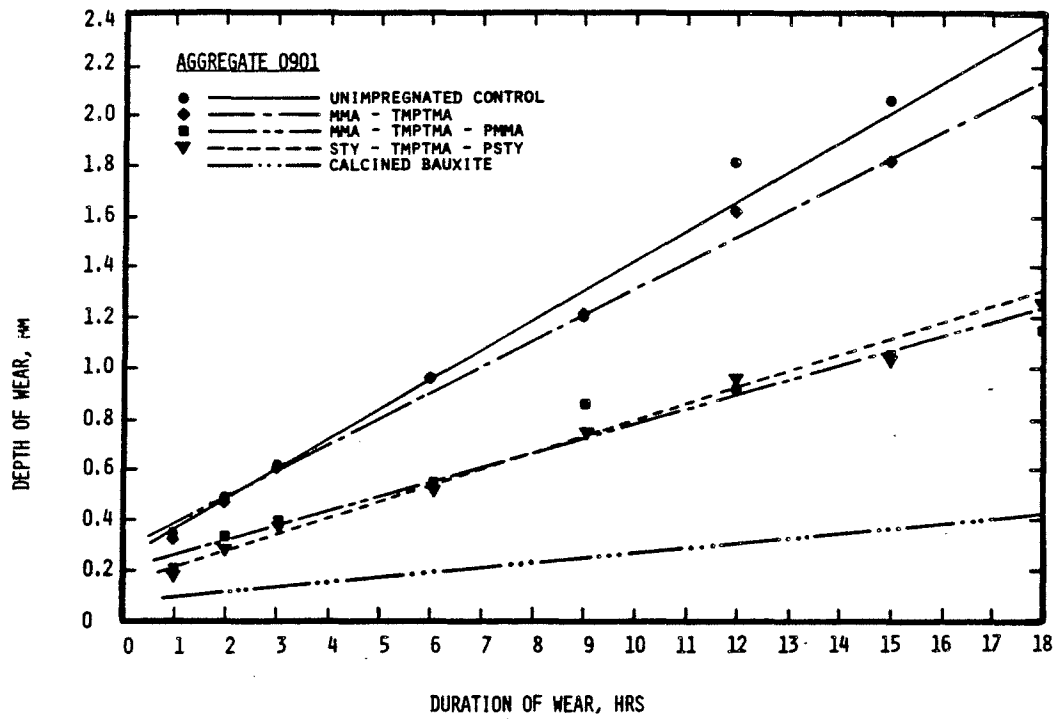


Figure 4 Extended Polish Wheel Tests - Aggregate 0901.

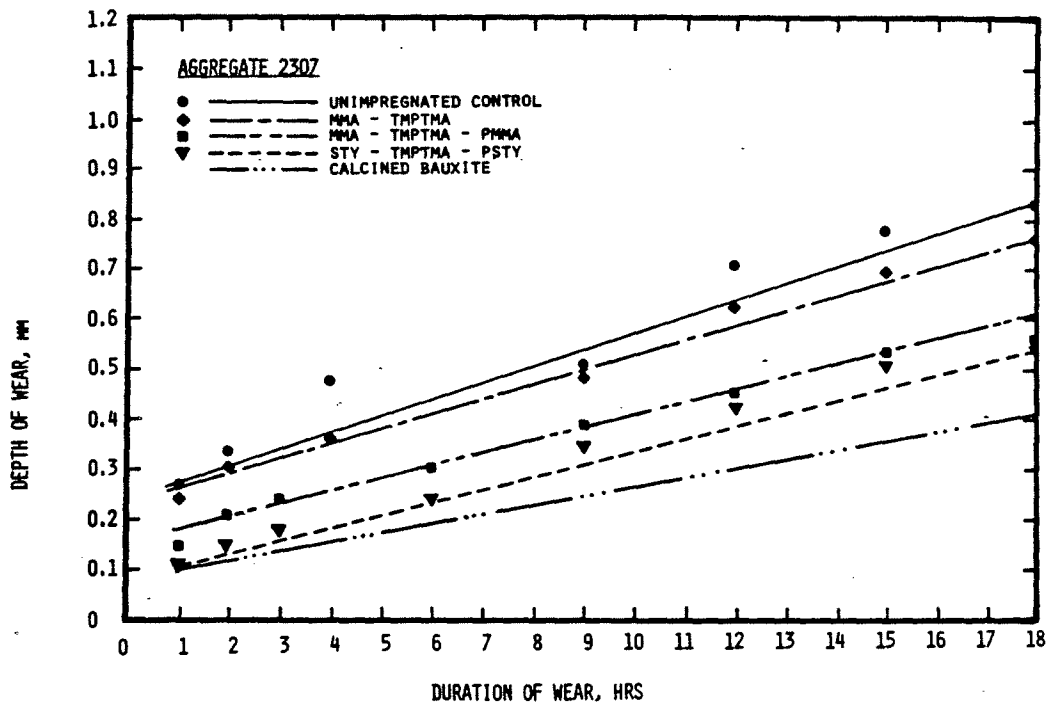


Figure 5 Extended Polish Wheel Tests - Aggregate 2307.

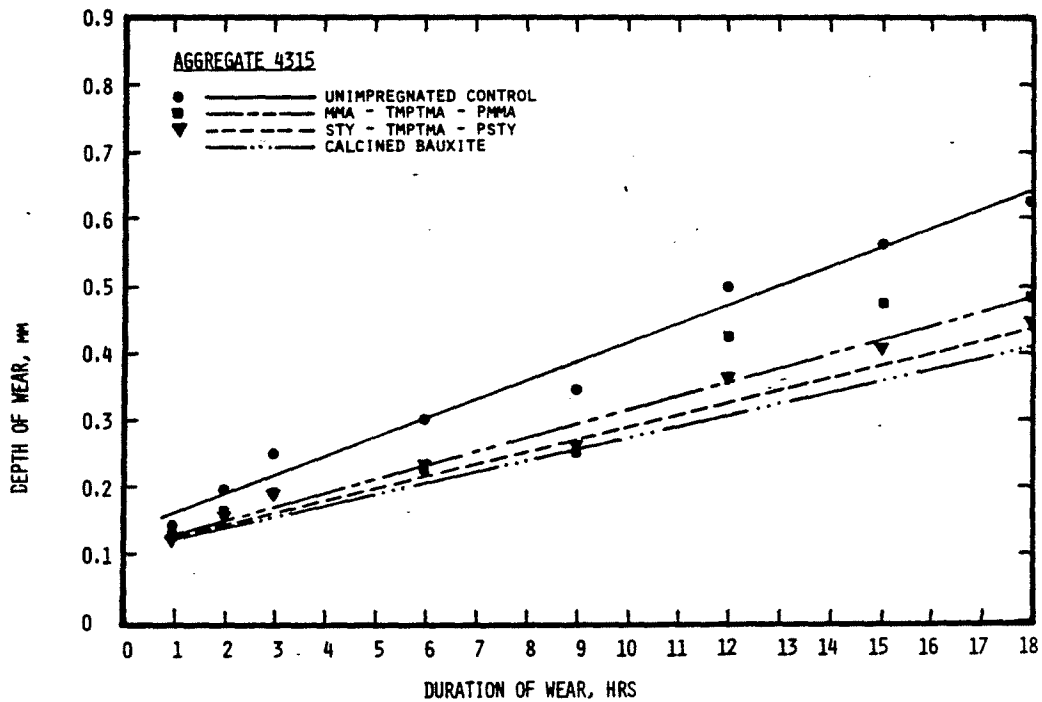


Figure 6 Extended Polish Wheel Tests - Aggregate 4315.

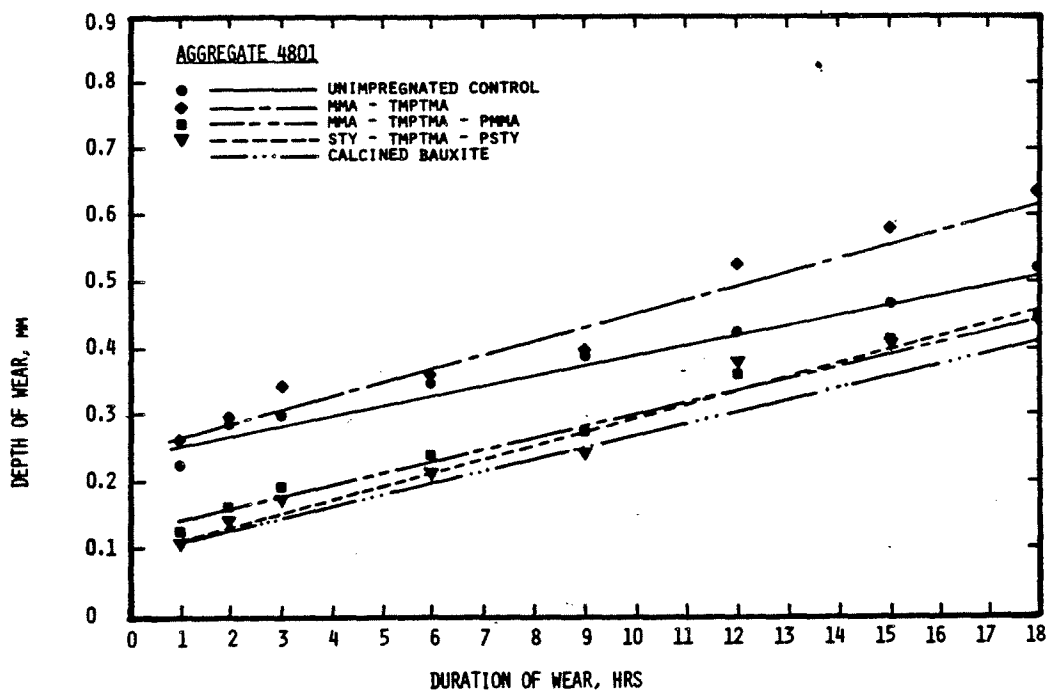


Figure 7 Extended Polish Wheel Tests - Aggregate 4801.

The reduction in polish resistance tended to vary directly with the viscosity of the impregnant, being the least with the lowest viscosity impregnant, the MMA-TMPTMA system. Because of its very low viscosity, some of this impregnant probably drained from the pore volume before it could be polymerized so that the pore volume was lined rather than filled, and thus the vesicular nature of the aggregate was maintained.

To a limited extent, improvements in wear resistance also tended to vary with impregnant viscosity. Over the first 9 hr of the polish wheel tests, they varied directly, i.e., the higher the viscosity the greater the improvement. However, over the second 9 hr the improvements in wear resistance of the aggregates impregnated with the higher viscosity systems (the sodium silicate solutions) began to decrease, probably because these impregnants did not penetrate as deeply into the aggregate as those with lower viscosities. The MMA-TMPTMA-PMMA system produced the greatest improvements in wear resistance over the 18 hour test period.

Both sodium silicate systems tested improved wear resistance but not polish resistance.

Aggregate 2307 - Each of the three impregnants evaluated improved the wear resistance of Aggregate 2307, but two of them also significantly reduced the polish resistance. The MMA-TMPTMA system produced the least improvement in wear resistance, but more importantly, it did not reduce the polish resistance, probably because it, as with Aggregate 0901, lined the pore volume rather than filling it.

The MMA-TMPTMA-PMMA and Sty-TMPTMA-PSty systems produced similar improvements in wear resistance, with the styrene-based system showing slightly better results. Both impregnants left coatings on the surface of the aggregate, as indicated by the reduction in the initial PV's. Both also produced significant reductions in the 9 hr PV's, probably (as with Aggregate 0901) due to filling of the pore volume with these higher viscosity impregnants, which (after curing) apparently have rates of wear very similar to those of the constituent minerals in the aggregate.

Aggregate 4315 - The two impregnants evaluated improved the wear resistance of Aggregate 4315 without lowering its polish resistance. Both the initial and the 9 hr PV's show no difference in the polish resistance of the unimpregnated and impregnated aggregates. The Sty-TMPTMA-PSty system produced a greater improvement in wear resistance than did the MMA-TMPTMA-PMMA system.

Aggregate 4801 - The effects of impregnation on the wear resistance and polish resistance of Aggregate 4801 were varied. Two of the three impregnants evaluated, the MMA-TMPTMA-PMMA system and the Sty-TMPTMA-PSty system, resulted in reductions in wear. The third impregnant, the MMA-TMPTMA system, did not improve the wear resistance, but in fact, reduced it for reasons not readily apparent. The only impregnant having any adverse effect on polish resistance was the Sty-TMPTMA-PSty system, as evidenced by the reduction in the 9 hr PV.

4.3.2 FHWA Microtexture Study

A limited study was done with the assistance of the Materials Division of the Federal Highway Administration, Office of Research to evaluate the effect of impregnation on aggregate surface microtexture as it relates to aggregate frictional characteristics. The surface microtexture characteristics of selected aggregate particles in polish wheel test coupons were measured with the FHWA's automated image analysis system located at the Fairbank Highway Research Station, Washington, DC, before and after the coupons were run on the polish wheel for 9 hr. Measurements were made on each of the four test aggregates and calcined Guyana bauxite (Aggregate 5101). Two impregnants were used, the MMA-TMPTMA-PMMA system and the Sty-TMPTMA-PSty system. These impregnants were selected on the basis of the polish wheel test results, which showed that they generally produced the best improvements in wear resistance.

One polish wheel coupon was used for each of the thirteen materials evaluated. Microtexture measurements were taken on at least five aggregate particles in each coupon, seven profiles being measured on each selected particle before and after the coupon was run on the polish wheel. The characteristics measured were average asperity density, average asperity height, and shape factor, the last being the product of the first two. The test results are summarized in Table 9.

The action of the polish wheel generally decreases both the average asperity density and the average asperity height (and therefore the shape factor). The decrease in asperity height is due to wearing away of the tops of individual mineral grains. The decrease in asperity density is due to

smoothing of the grain surfaces, which eliminates some of the smaller peaks occurring as bumps on the sideslopes of the grains. These effects are influenced by the initial characteristics of the aggregate such as grain size, hardness of minerals, bonding of the grains, etc., all of which must be considered in evaluating the results of this study. The test results for each of the four aggregates are presented below.

Aggregate 0901 - The shape factor and average asperity height were lower for each sample after polishing. The average asperity density gave results that were less clear-cut, since that for the unimpregnated aggregate was significantly higher after polishing. The variable porosity of the aggregate may account for this discrepancy. The stones picked for measurement in the coupon made with the unimpregnated aggregate were highly porous; therefore, although their rate of wear may be high, they would tend to maintain a good microtexture. On the other hand, the stones picked for measurement in the coupons made with the MMA-TMPTMA-PMMA and the Sty-TMPTMA-PSty impregnated aggregates were much denser and would tend to polish, although they would wear at a lower rate. Any benefit derived from impregnation appears to be masked by the variation in porosity of the aggregate particles evaluated.

Aggregate 2307 - For both the unimpregnated and impregnated aggregates, the shape factors and average asperity heights were lower after polishing, but the average asperity density was slightly higher. For the impregnated aggregates, the reason for the increased asperity density may be that some of the impregnant filled in the natural roughness of the aggregates prior to polishing, and the action of the polish wheel removed this filling in effect renewing the surface.

Aggregate 4315 - The measurements showed few changes due to the action of the polish wheel. For the unimpregnated sample, the average asperity density stayed about the same but the average asperity height increased; therefore the shape factor increased. Since the surface of the unpolished aggregate was indeed smoother than the surface of the same aggregate after polishing, the action of the polish wheel was probably removing the softer mineral matter around the quartz grains. The increased exposure of the quartz grains is reflected in the increased average asperity height after polishing. The results for the MMA-TMPTMA-PMMA impregnated aggregate were similar, but the average asperity height increase was less pronounced, perhaps because the impregnant was holding some of the soft material in place.

The Sty-TMPTMA-PSty impregnated aggregate showed a decrease in average asperity height and a significant increase in average asperity density. As with Aggregate 2307, this may indicate the presence of some impregnant on the surface which the polishing removed. Also, this impregnant may be more effective than the MMA-TMPTMA-PMMA system in holding in place the softer material between the quartz grains; this would tend to cause polishing to decrease rather than increase the average asperity height.

Aggregate 4801 - For both the unimpregnated and impregnated aggregates, the average asperity density was decreased by polishing, but the average asperity height either remained about the same or increased. As with

Table 9 Microtexture Study Test Results

Aggregate Code No.	Impregnant ^a	Before Polishing				After Polishing			
		Average Asperity Density, Peaks/ μ	Average Asperity Height, μ	Shape Factor ^b	PV ^c	Average Asperity Density, Peaks/ μ	Average Asperity Height, μ	Shape Factor	PV
0901	--	0.0039	38.8	0.151	50	0.0047	29.4	0.139	41
	MMA	0.0037	35.2	0.130	42	0.0040	24.4	0.097	31
	Sty	0.0039	32.2	0.124	45	0.0030	25.8	0.076	31
2307	--	0.0039	30.7	0.118	48	0.0044	25.2	0.112	32
	MMA	0.0038	32.3	0.123	40	0.0041	20.6	0.084	29
	Sty	0.0032	30.9	0.100	45	0.0038	24.8	0.095	31
4315	--	0.0046	24.8	0.115	41	0.0044	29.2	0.129	35
	MMA	0.0043	27.8	0.121	42	0.0043	29.9	0.129	36
	Sty	0.0034	34.4	0.116	43	0.0043	30.5	0.123	34
4801	--	0.0038	39.2	0.147	53	0.0031	45.2	0.140	43
	MMA	0.0037	40.7	0.149	48	0.0031	39.4	0.122	42
	Sty	0.0040	36.5	0.145	46	0.0031	40.3	0.127	39
5101	--	0.0041	39.4	0.163	50	0.0037	42.4	0.159	38

^aMMA = 83 wt% methyl methacrylate - 5 wt% trimethylolpropane trimethacrylate - 12 wt% poly(methyl methacrylate); Sty = 57 wt% styrene - 38 wt% trimethylolpropane trimethacrylate - 5 wt% polystyrene; dash indicates unimpregnated control.

^bShape factor = average asperity density x average asperity height.

^cPV = polish value. Polish wheel run for 9 hr.

Aggregate 4315, the polishing action probably removed softer material from around the harder mineral grains of the unimpregnated aggregate, thereby increasing the texture. This appears to apply also to the impregnated aggregate, but to a lesser extent because of the impregnants' ability to hold the softer material in place.

4.3.3 Maryland Circular Test Track Study

A study was carried out at the Maryland State Highway Administration's circular test track to evaluate the effect of impregnation on the polish resistance of three of the aggregates: Aggregates 0901, 4315 and 4801. (Supply limitations precluded the testing of Aggregate 2307.) Two impregnants were used, the MMA-TMPTMA-PMMA system and the Sty-TMPTMA-PSty system, selected on the basis of the polish wheel test results.

Polish resistance was evaluated by measuring the frictional resistance of the test specimen with the British Portable Tester, and by determining the polish strain value from the frictional resistance offered by the specimens to the slippage of a test tire over their surfaces.

The aggregates were tested under simulated traffic conditions for a total of 2,000,000 wheel passes, with frictional measurements taken at intervals to evaluate the change in polish resistance with wheel passes. The terminal polish resistance value was predicted for each aggregate by fitting the test data to a mathematical model based on the theory that as the number of wheel passes approaches infinity the polish resistance approaches a terminal value.

The control aggregate for the test track was also included in the study to provide a means for correlating the data. The control aggregate was a dolomitic marble with relatively low polish resistance. It has a BPN of 28, a polish strain value of 6, and a PV of 27 on the British polish wheel.

The values obtained for terminal polish resistance, as measured by the British Portable Tester, and for polish strain are listed in Table 10. In general, neither of the impregnants produced any significant change in the polish resistance of any of the aggregates. With only two exceptions, the BPN and the polish strain value for the impregnated aggregate remained within ± 1 of the values for the unimpregnated aggregate. A variation of ± 1 is within the normal range of experimental error.

Figures 8 through 10 show the variation in BPN and polish strain value with number of wheel passes for the three aggregates. They are plots of the raw, unadjusted data points from which the terminal BPN and polish strain values were calculated. The BPN value tends to drop off rapidly during the initial stages of testing before leveling off. The polish strain value shows a less defined pattern of variation, appearing to oscillate before approaching a final value.

Table 10 Polish Resistance Test Results - Maryland
Circular Test Track Study

Aggregate Code No.	Impregnant ^a	BPN ^b	Polish Strain Value
0901	—	27	7
	MMA	26	6
	Sty	28	6
4315	—	46	13
	MMA	46	11
	Sty	45	12
4801	—	62	19
	MMA	64	19
	Sty	62	18
Control ^c	—	28	6

^aDefined in Table 9.

^bBPN = British Portable number, as measured by the British Portable Testor.

^cControl = control aggregate for test track.

4.3.4 North Carolina Circular Test Track Study

A study was carried out at the North Carolina Department of Transportation circular test track to evaluate the effect of impregnation on the polish resistance of the four test aggregates. Two impregnants were used, the MMA-TMPTMA-PMMA system and the Sty-TMPTMA-PSty system, selected on the basis of the polish wheel test results.

Polish resistance was evaluated by measuring the frictional resistance of the test specimen with the British Portable Tester and the Variable Speed Friction Tester (VST). The test specimens were polished for 8 hr, with frictional measurements made at intervals to evaluate the change in polish resistance with time.

The control aggregate for the test track was also included in the study to provide a reference for correlating the data collected. The control aggregate was a quartzite disc muscovite with medium to low polish resistance characteristics, having a PV of 30.

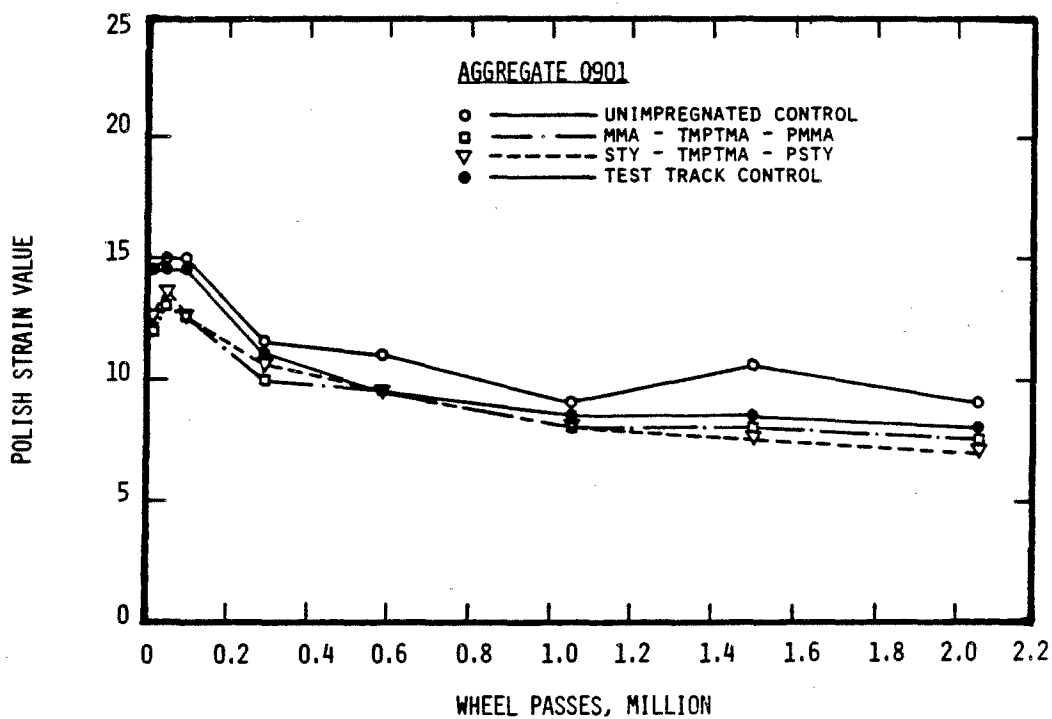
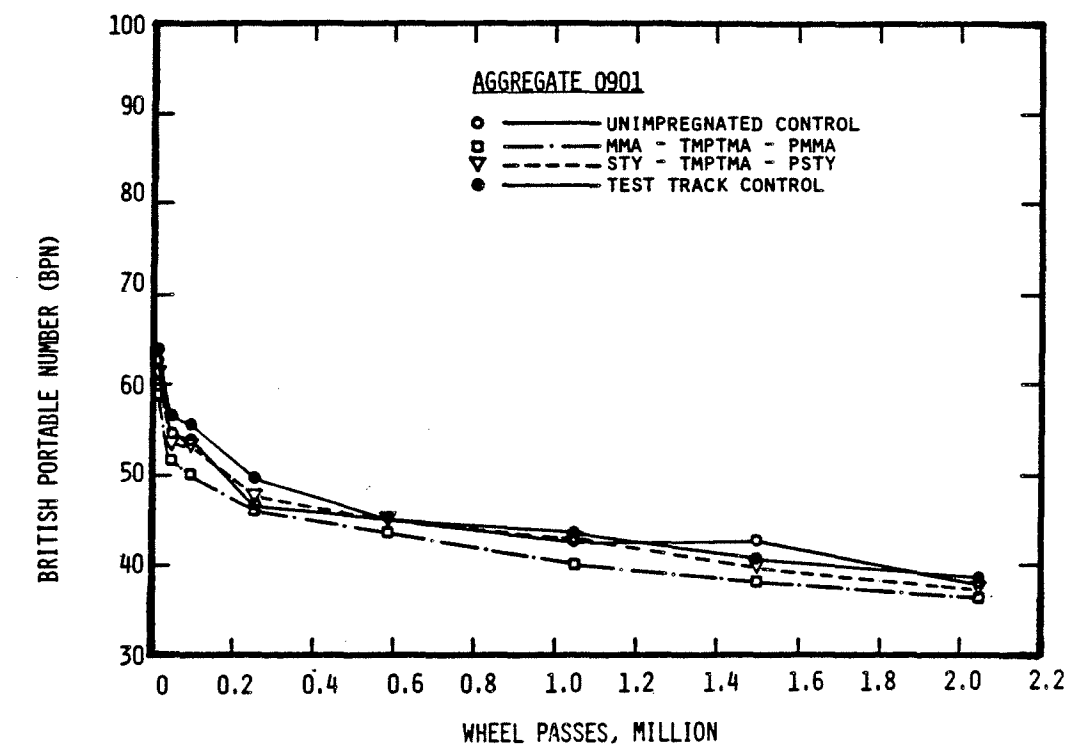


Figure 8 Maryland Circular Test Track Results - Aggregate 0901.

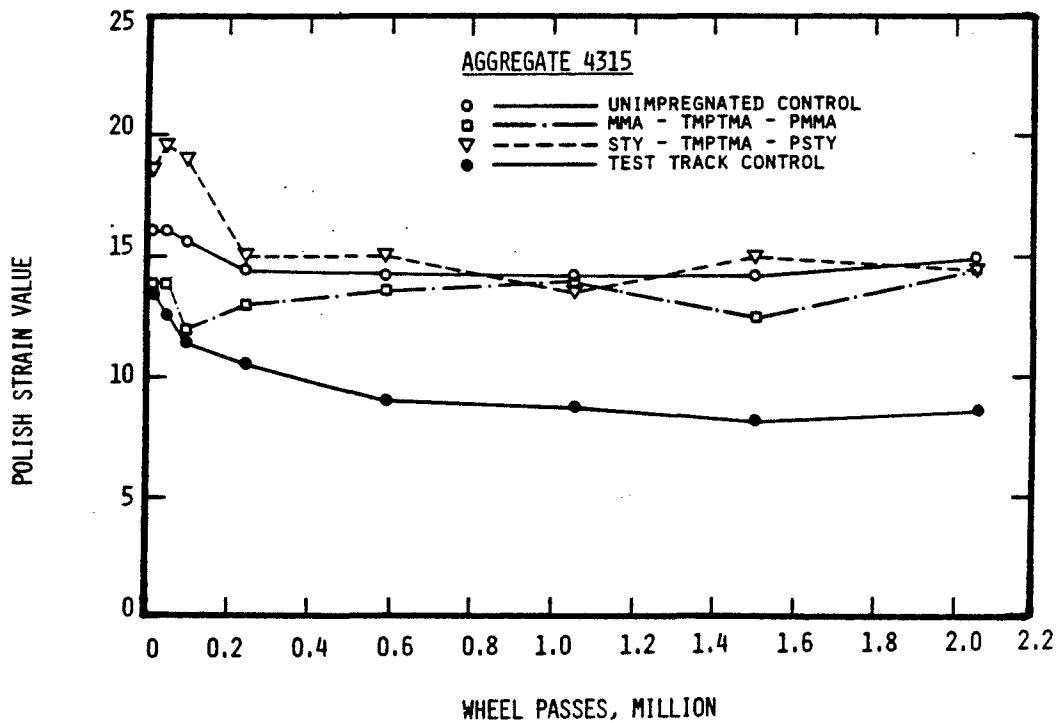
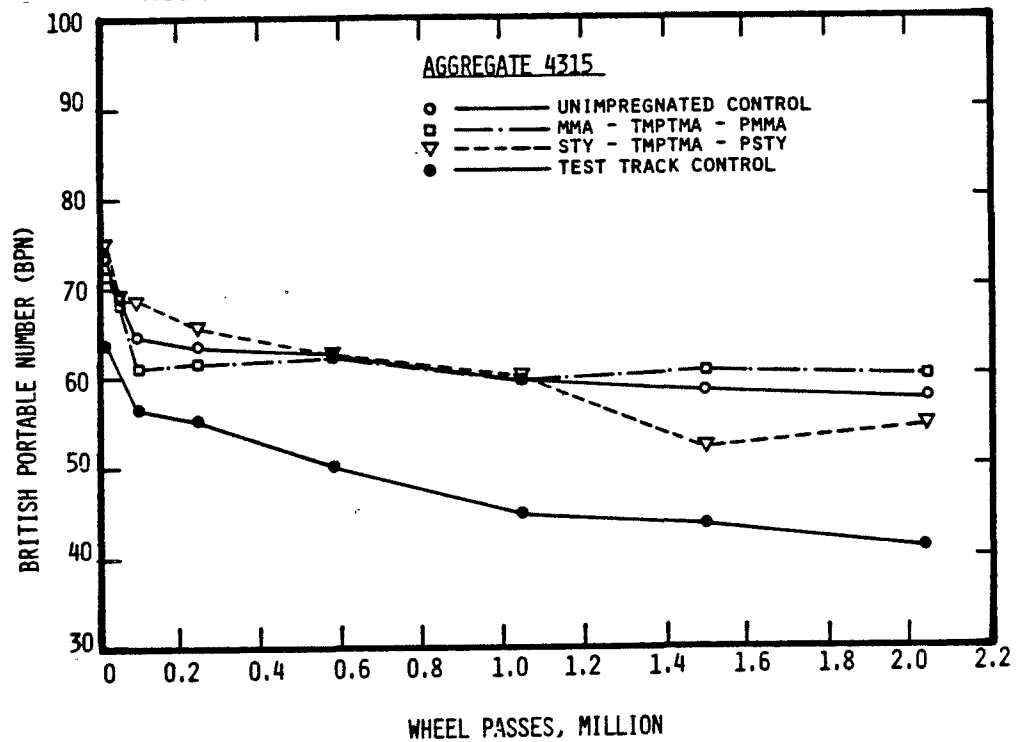


Figure 9 Maryland Circular Test Track Results - Aggregate 4315.

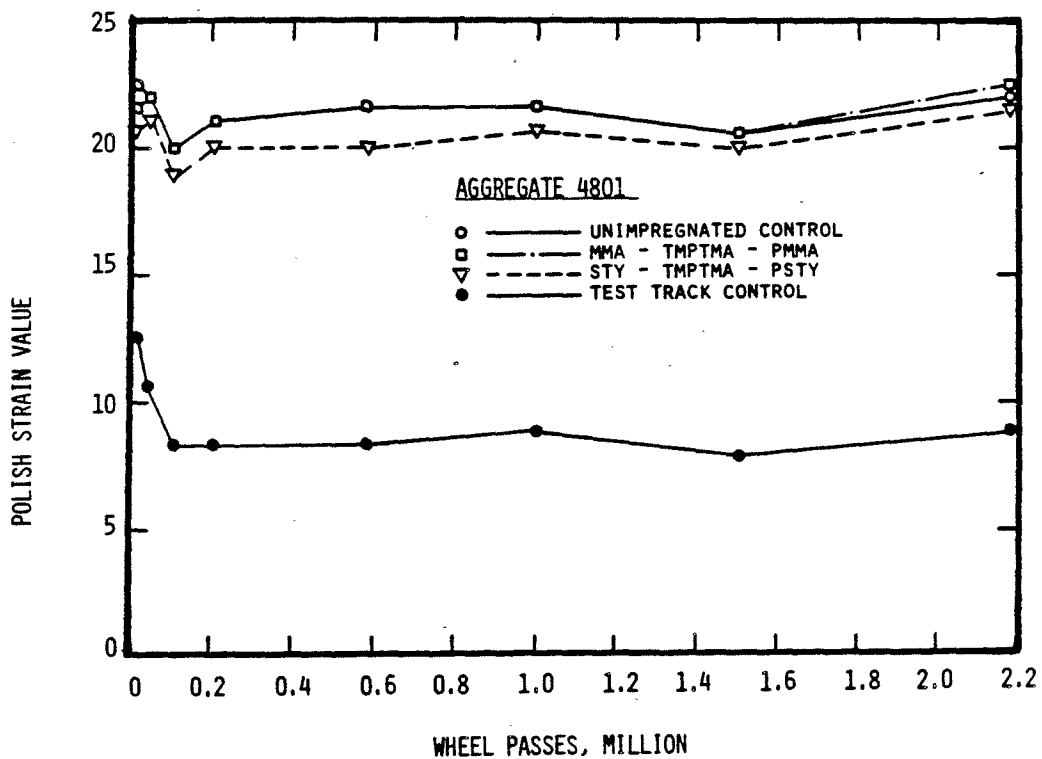
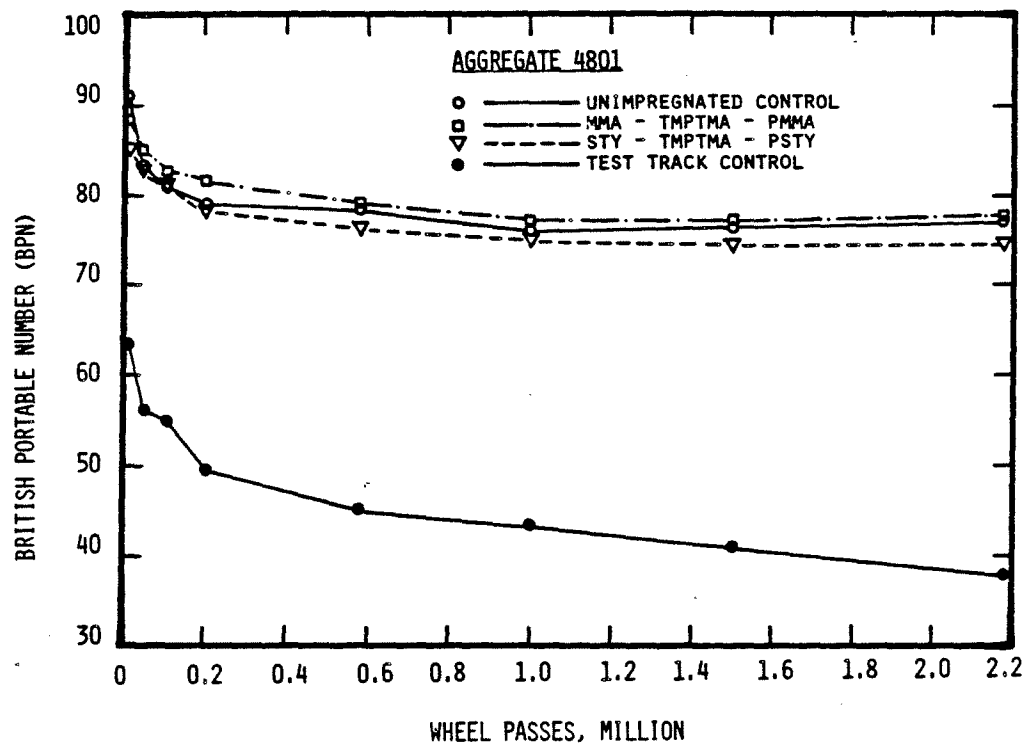


Figure 10 Maryland Circular Test Track Results - Aggregate 4801.

The purpose of the test track study was to compare the polish resistance of unimpregnated and impregnated aggregates, but the data obtained also provides an indication of how the various aggregates will perform in the field. The North Carolina DOT has done considerable research on correlating polish resistance data obtained in the field using the VST with data obtained using their skid trailer. The results have shown excellent correlation for test speeds of 30, 40, and 50 mph. Results have also shown that field polish seldom exceeds the circular test track polish obtained at 3 to 4 hr.

Variable Speed Test Numbers (VSN) and British Portable Numbers (BPN), measured at 4 hr, are summarized in Table 11. VSN were measured for simulated test speeds of 8, 30, 40, and 50 mph, and BPN for 8 mph. The results can be summarized as follows.

Aggregate 0901 - Reductions were noted in the VSN for both the MMA and the Sty impregnated aggregates, with the Sty impregnated aggregate showing greater reductions. Essentially no difference was found between the BPN of the unimpregnated and impregnated aggregates.

Aggregate 2307 - Reductions were noted in both VSN and BPN for the MMA impregnated aggregate. The Sty impregnated aggregate showed slight reductions in VSN values at 8 and 30 mph but not at 40 and 50 mph, and slight reduction in the BPN.

Table 11 Polish Resistance Test Results - North Carolina Test Track Study

Aggregate Code No.	Impregnant ^a	VSN ^b				BPN ^c at 8 mph
		Test Speed, mph				
		8	30	40	50	
0901	---	46.0	42.0	39.2	37.3	48.8
	MMA	41.1	39.5	37.8	36.7	48.1
	Sty	39.5	37.0	35.3	33.8	49.1
2307	---	41.8	39.5	38.3	36.8	52.0
	MMA	39.5	36.8	35.8	34.4	50.5
	Sty	40.3	38.5	38.0	36.8	50.8
4315	---	45.9	44.3	42.6	41.1	55.0
	MMA	45.0	42.9	41.2	39.7	55.5
	Sty	47.4	45.5	43.8	42.3	54.9
4801	---	45.5	43.0	42.0	41.0	58.2
	MMA	46.1	43.1	41.5	39.9	56.2
	Sty	45.4	43.2	41.8	40.0	58.0
Control		45.6	43.3	41.8	40.2	53.1

^aDefined in Table 9.

^bVSN = Variable Speed Test Number.

^cBPN = British Portable Number

Aggregate 4315 - The MMA and Sty impregnated aggregates showed slight reduction in the VSN, but the BPN remained essentially unchanged.

Aggregate 4801 - The VSN were essentially unchanged for both the MMA and the Sty impregnated aggregate at test speeds of 8 and 30 mph and were slightly reduced at 40 and 50 mph. The BPN remained essentially unchanged for the Sty impregnated aggregate and was reduced for the MMA impregnated aggregate.

Aggregates 4315 and 4801, unimpregnated and impregnated, generally had VSN and BPN equal to or better than those for the test track control aggregate.

The variation in VSN with time, measured at 40 mph, is presented for each aggregate in Figures 11 through 14. The results can be summarized as follows.

Aggregate 0901 - Except for the initial 4 hr values, the VSN for both the MMA impregnated and Sty impregnated aggregates were consistently greater than those of the unimpregnated aggregate. The data also indicate that the VSN for the impregnated aggregates are relatively constant over the last 4 hr of the test, while the VSN for the unimpregnated aggregate continue to decrease.

Aggregate 2307 - The VSN for both the MMA impregnated and the Sty impregnated aggregates were consistently lower than that of the unimpregnated aggregate.

Aggregate 4315 - The VSN for both the MMA impregnated and the Sty impregnated aggregates were consistently lower than that of the unimpregnated aggregate.

Aggregate 4801 - Generally, no significant difference was seen in the VSN for the unimpregnated and for the impregnated aggregate over the duration of the test.

In general, the study indicated that the polish resistance of the aggregates was not improved by impregnation and in some cases was lowered.

4.4 Evaluation of Impregnated Aggregates in Pavement Systems

4.4.1 Concrete Mixes

Tests were done to evaluate the strength of concrete cast with unimpregnated and impregnated coarse aggregate. The properties evaluated were compressive, flexure, and splitting tensile strength. In addition, a limited number of cylinders were tested for freeze-thaw durability by determining the compressive strength at 0, 25, and 50 cycles.

Concrete test specimens were cast with each of the four aggregates, unimpregnated and impregnated with the MMA-TMPTMA-PMMA, and with one aggregate impregnated with the Sty-TMPTMA-PSty. The test results, presented in Table 12 may be summarized as follows.

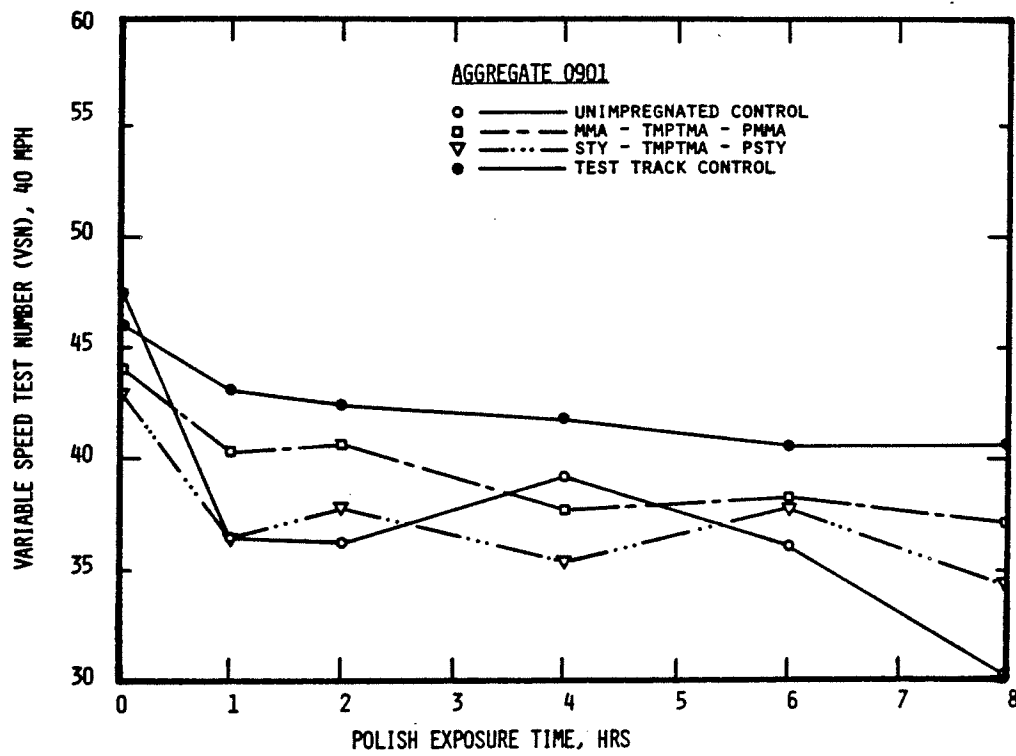


Figure 11 North Carolina Test Track Results - Aggregate 0901.

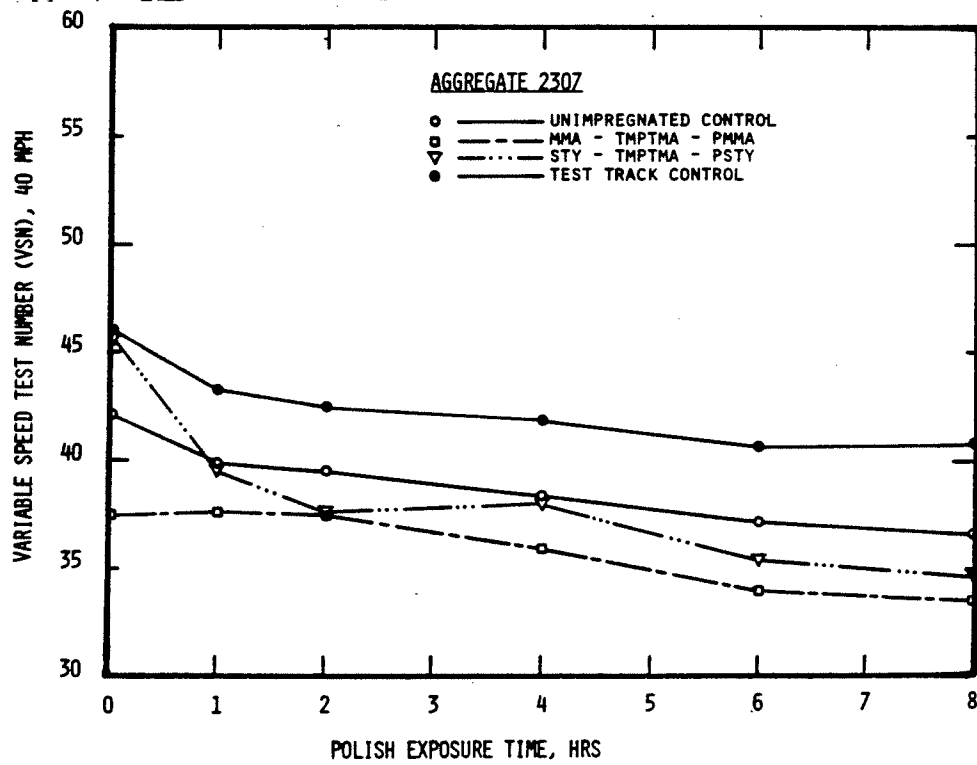


Figure 12 North Carolina Test Track Results - Aggregate 2307.

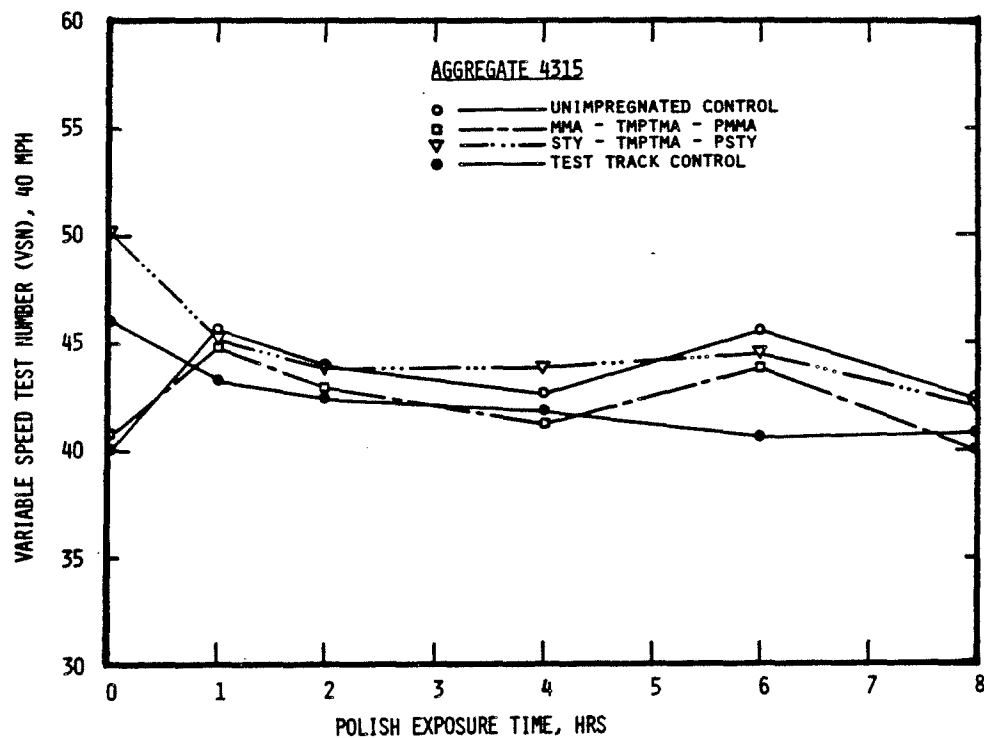


Figure 13 North Carolina Test Track Results - Aggregate 4315.

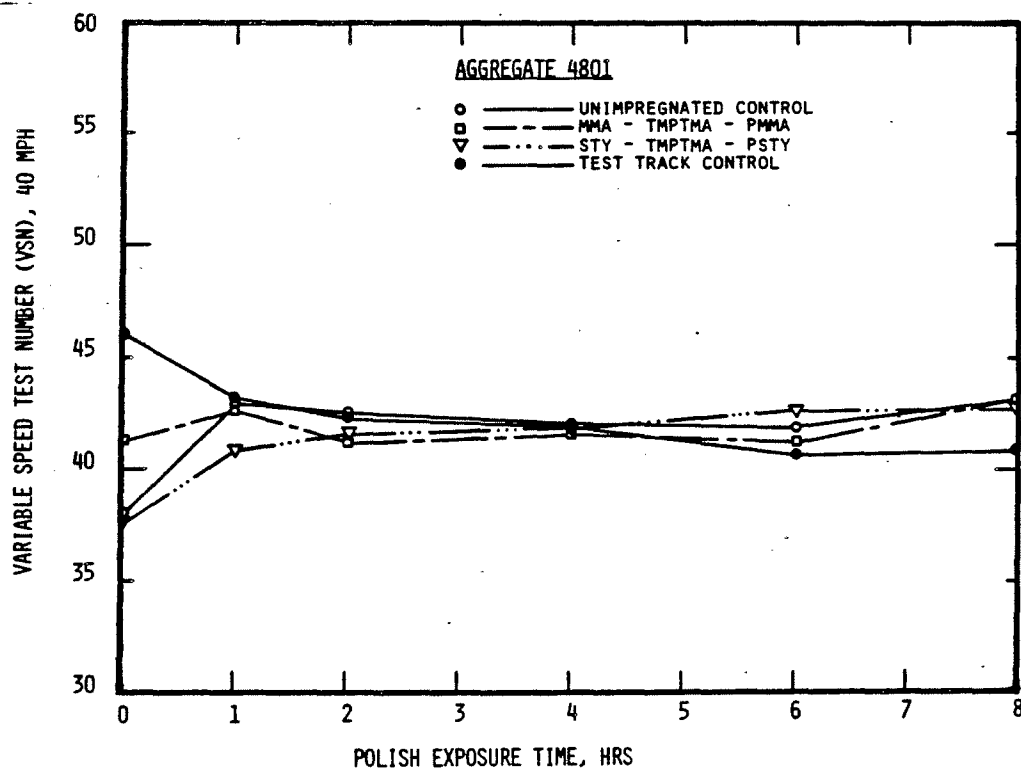


Figure 14 North Carolina Test Track Results - Aggregate 4801.

Table 12 Strength of Concrete Cast with Unimpregnated and Impregnated Aggregate

Aggregate No.	Impregnant ^a	Polymer Loading, wt%	Compressive Strength, MPa (psi)	Flexure Strength, MPa (psi)	Splitting Tensile Strength, MPa (psi)
0901	---	0	25.2 (3660)	2.9 (420)	2.6 (380)
	MMA	3.62	21.1 (3060)	3.0 (430)	2.9 (420)
2307	---	0	33.3 (4830)	4.1 (590)	3.3 (480)
	MMA	1.99	28.0 (4070)	4.3 (630)	3.4 (500)
4315	---	0	22.3 (3230)	2.5 (360)	2.6 (380)
	MMA	2.04	18.1 (2620)	2.5 (370)	2.2 (320)
4801	---	0	23.0 (3340)	3.8 (550)	2.3 (340)
	MMA	1.72	25.1 (3650)	4.0 (580)	2.9 (420)
	Sty	1.73	25.8 (3740)	4.3 (620)	3.0 (430)

^aDefined in Table 9.

Aggregate 0901 - Two of the three strength properties were improved by using the MMA-TMPTMA-PMMA impregnated aggregate. The flexure strength was increased by 2% and the splitting tensile strength by 11%. The compressive strength was reduced by 16%.

Aggregate 2307 - Two of the three properties were improved by using aggregate impregnated with the MMA-TMPTMA-PMMA system. The flexure strength was increased by 7% and the splitting tensile strength by 5%. The compressive strength was reduced by 16%.

Aggregate 4315 - Two of the three properties were reduced by using the MMA-TMPTMA-PMMA impregnated aggregate. The compressive strength was reduced by 19% and the splitting tensile strength by 14%. The flexure strength increased by 4%.

Aggregate 4801 - All three of the properties were improved by using either the MMA-TMPTMA-PMMA or the Sty-TMPTMA-PSty impregnated aggregate. The concrete cast with the MMA-based system showed a 9% increase in compressive strength, a 6% increase in flexure strength, and a 25% increase in splitting tensile strength. The concrete cast with the aggregate impregnated with the Sty-based system showed a 12% increase in compressive strength, a 13% increase in flexure strength, and a 27% increase in splitting tensile strength.

The reduction in strength of three of the five concrete mixes cast with impregnated aggregate may be due in part to the presence of residual polymer on the aggregate surface, which could interfere with the aggregate-matrix bond, or to the lack of modification of the concrete mix proportions to take into account the differences in the specific gravity and water absorption between the unimpregnated and impregnated aggregates. The concrete mix proportions for each aggregate group were based on the properties of the unimpregnated aggregates. It is not readily apparent, however, why concrete cast with a particular impregnated aggregate would exhibit increases in some properties and decreases in others.

Table 13 shows the results of the freeze-thaw tests, which were done on concrete cast with Aggregate 4801 impregnated with the MMA-TMPTMA-PMMA system and the Sty-TMPTMA-PSty system. The concrete cast with unimpregnated aggregate showed a significant reduction (13%) in compressive strength after 50 freeze-thaw cycles, whereas the concretes cast with impregnated aggregates showed essentially no reduction. The improvement in freeze-thaw durability may be due to the improved strength of the individual aggregates resulting from the interconnecting network of polymer developed throughout the pore system. This internal network reinforces the aggregate, allowing it to withstand the hydraulic pressures developed within the pore system by the water being expelled during freezing, and thus prevents the aggregate from fracturing and thereby weakening the concrete.

Table 13 Freeze-thaw Test Results for Concrete Cast with Aggregate 4801

Impregnant ^a	Polymer Loading, wt%	Compressive Strength, MPa (psi)		
		Number of Freeze-Thaw Cycles		
		0	25	50
---	0	23.0 (3340)	20.8 (3020)	19.9 (2890)
MMA	1.72	25.1 (3650)	24.7 (3580)	25.5 (3700)
Sty	1.73	25.8 (3740)	24.6 (3570)	25.6 (3720)

^aDefined in Table 9.

4.4.2 Bituminous Mixes

The static immersion stripping test (ASTM D 1664) was done to evaluate the affinity of AC-20 asphalt to bind to unimpregnated and impregnated aggregates. Each of the four test aggregates, impregnated with the MMA-TMPTMA-PMMA system and the Sty-TMPTMA-PSty system, was evaluated, and three aggregates with known stripping characteristics were tested as controls.

The results, summarized in Table 4.9, show no evidence of stripping for either the unimpregnated or impregnated aggregates after the prescribed 16 to 18 hr of immersion in distilled water at 25°C (77°F). Although not part of the standard test procedure, the aggregates were tested for an additional 16 to 18 hr in distilled water at 60°C (140°F). This more severe test did induce some stripping in each of the aggregates. For Aggregates 0901 and 2307, which appear to be naturally good non-stripping aggregates, the impregnants, particularly the Sty-TMPTMA-PSty, increased the tendency strip. For Aggregates 4315 and 4801, which appear to be prone to stripping, the impregnants reduced the tendency to strip.

Table 14 Static Immersion Stripping Test Results

Aggregate Code No.	Impregnant ^a	Appearance Immed.	After 16-18 hr in water at 77°F, Percent Coated	Add'l 16-18 hr in water at 140°F, Percent Coated
		After Coating and Immersion, Percent Coated		
0901	---	100	100	95+
	MMA	100	100	95+
	Sty	100	100	80-90
2307	---	100	100	95+
	MMA	100	100	95+
	Sty	100	100	85-95
4315	---	100	100	10-30
	MMA	100	100	95+
	Sty	100	100	75-85
4801	---	100	100	55-65
	MMA	100	100	70-80
	Sty	100	100	75-85
C1 ^b		100	100	40-60
C2		100	100	95+
C3		100	100	75-85

^aDefined in Table 9.

^bControl aggregates: C1 = Chantilly diabase; C2 = Riverton limestone;
C3 = White marsh gravel.

5.0 PRELIMINARY IMPREGNATION PROCESS DESIGN AND ECONOMIC EVALUATION

The research reported here has shown that the physical and mechanical properties of natural aggregates can be significantly improved by impregnation, but economic considerations will ultimately determine the feasibility of the process. At present, the most promising application of impregnation appears to be in beneficiating marginal aggregates. The supply of quality aggregates in many regions of the country is being rapidly depleted. As a result, these regions are having to import large quantities of quality aggregates. However, in most instances, these regions have large supplies of marginal aggregates that could be beneficiated by impregnation and thus eliminate the need for importation. The choice between beneficiating and importing will depend solely on economics. Assuming that the costs of excavation and production are about equal for marginal and quality aggregates, the choice between the alternatives will depend on the cost of impregnating marginal aggregate versus the cost of importing quality aggregate.

The cost of impregnation includes material costs, which will vary depending on the impregnant selected and on the loading requirements of the aggregate to be impregnated; direct operating costs, which include such items as utilities; maintenance and repair costs; labor costs; and capital recovery of investments.

The cost of importation include the direct cost of shipping the aggregate by either truck or railroad, and handling costs for loading and unloading. Shipping costs may include fuel surcharges to adjust for rising fuel prices.

The initial investment costs for an impregnation facility will depend primarily on anticipated production requirements and location. The ideal location for the facility would be at the aggregate processing plant, so that the impregnation would be just another step in the overall production process. Figure 15 lists the steps in the manufacture of impregnated mineral aggregates.

The processing scheme can be summarized as follows. The excavated raw material first undergoes preliminary crushing and is then conveyed to the scrubbers for removal of fines adhering to the material. Next it is sent to the secondary crushers for final sizing. Normally the material would then be screened and sieved to a specified grading, but this is the point where marginal aggregate will be impregnated.

After passing through the secondary crushers, the raw material is dried to remove all the free moisture within the pore volume. In some cases it may be necessary to rewash the material after going through the secondary crushers if it contains excessive clay and dirt. After drying, the material must be allowed to cool to a maximum temperature of 38°C (100°F) before it can be impregnated. It is then put into the impregnation vessel and saturated with the impregnant, which is subsequently cured in situ. The impregnated aggregate is screened and sieved to a specified grading and stockpiled.

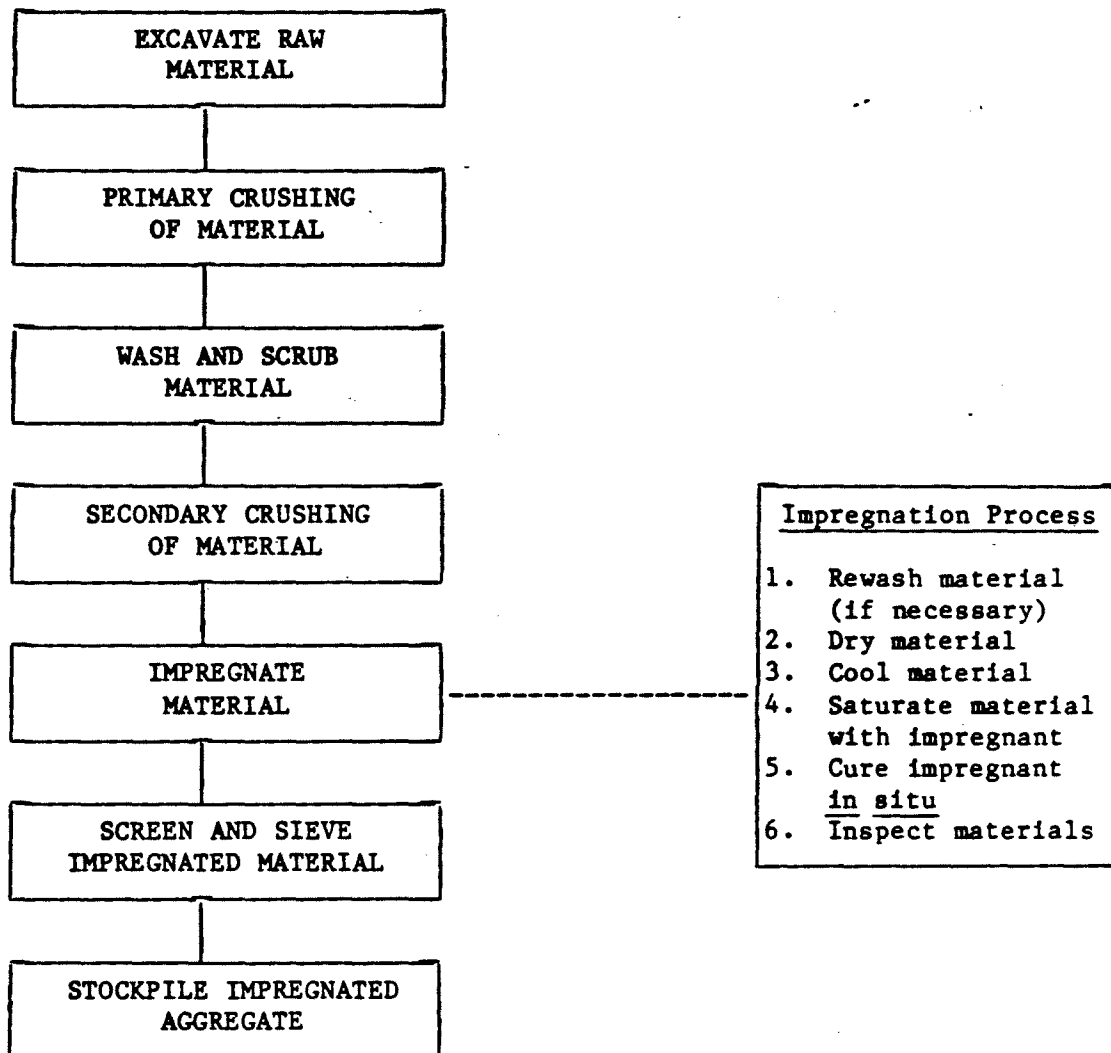


Figure 15 Production Scheme for the Manufacture of Impregnated Mineral Aggregates.

Except for the impregnation vessel, which may require a special design, most of the equipment and technology needed to construct a facility are readily available. Dryers normally used in the production of hot-mix asphalt can be modified for use in drying the aggregate before impregnation and cooling it with cold air. In the case of water-based impregnants such as sodium silicate, dryers can be used for the curing which consists simply of driving off the water. Design of the impregnation vessel will depend on specific details of the impregnation process such as the degree of evacuation, the soaking time, the amount of pressurization, and the method of curing.

No attempt is made here at a detailed analysis of the costs of an impregnation facility, because of the large number of factors to be considered, and because of the rapidly changing nature of the economy. A preliminary estimate of the investment and production costs was made based on work done at BNL in 1967 on the design of an impregnation facility for producing polymer impregnated concrete pipe (14). At that time the initial investment was estimated at \$400,000; with an annual inflation rate of 7%, this would now be about \$764,000. A breakdown of estimated production costs is presented in Table 15, and the material costs for the impregnants used in this study are summarized in Table 16. These data can be used to make a preliminary estimate of the production costs if the amount of aggregate that can be impregnated on a daily, monthly, or annual basis is known. For example, since the cost of the impregnant comprises about 23% of the total production costs, the cost of impregnating 1000 kg of aggregate to a loading of 2 wt% would be about \$150.69 with the MMA-TMPTMA-PMMA system and \$23.39 with sodium silicate.

For comparison, the cost of transporting 1000 kg of aggregate by truck is generally estimated to be about \$0.294/mile, and a 13 to 15% fuel surcharge is usually added to cover continually rising fuel prices. In practice it is economically impractical to transport aggregate more than 20 to 35 miles (15, 16) because transportation costs would begin to exceed the quarry price of the aggregate significantly.

Table 15 Breakdown of Production Costs for an Impregnation Facility

Production Cost	Percent of Total Production Cost
Raw Materials:	
Impregnant	23
Utilities	2
Labor and Maintenance:	
Labor and Overhead	42
Health Physics Control	3
Maintenance and Supplies	8
Indirect Costs:	
Depreciation	18
Taxes and Insurance	4
Total	100

Table 16 Material Costs of Impregnants

Impregnant	Unit Cost of Impregnant, \$/kg ^a	Impregnant Cost \$/1000 kg Agg. Impregnated ^b
MMA-TMPTMA ^c	1.611	32.22
MMA-TMPTMA-PMMA ^d	1.732	34.64
Sty-TMPTMA-PSty ^e	2.108	42.16
Phenol Formaldehyde	1.337	26.74
Sodium Silicate	0.269	5.38

^a\$1/kg \approx \$0.464/lb.

^bImpregnant loading = 2 wt%. 1000 kg \approx 1.11 ton.

^c90 wt% methyl methacrylate - 10 wt% trimethylolpropane trimethacrylate - 1 wt% silane - 1 wt% initiator.

^d83 wt% methyl methacrylate - 5 wt% trimethylolpropane trimethacrylate - 12 wt% poly(methyl methacrylate) - 1 wt% silane - 1 wt% initiator.

^e57 wt% styrene - 38 wt% trimethylolpropane trimethacrylate - 5 wt% polystyrene - 1 wt% silane - 1 wt% initiator.

6.0 CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

The following conclusions can be drawn from the results of the research described in this report.

1. The physical and mechanical properties of natural aggregates can be significantly improved by impregnation with the materials investigated.

2. Impregnation with low viscosity impregnants can significantly improve the wear resistance of both carbonate and siliceous aggregates, but not the polish resistance, which is reduced in carbonate aggregates and unchanged in siliceous aggregates. Thus impregnation appears to be a viable means of improving the wear resistance of aggregates that already contain the basic constituent minerals required for good polish resistance.

3. The use of impregnated aggregate can increase the strength of concrete, in some cases, and also its freeze-thaw durability.

4. Impregnation can reduce the tendency of some aggregates to strip.

6.2 Recommendations

Because of limited scope of the research described in this report, further work is needed so that the potential benefits of impregnating natural aggregates can be fully understood and utilized. This work should not be limited to improving the wear and skid resistance of natural aggregates. The significant upgrading of the physical and mechanical properties of the aggregates studied indicates that investigations should be continued on the impregnation of aggregates with poor mechanical or durability characteristics (e.g., low abrasion resistance, freeze-thaw deterioration, and susceptibility to D-cracking).

Additional work is needed to optimize the parameters of the impregnation process: evacuation and soaking times, pressurization requirements, and curing time.

Further work is needed on the mix design parameters, which are affected when impregnated aggregates are incorporated into concrete or bituminous mixtures.

The wear resistance and polish resistance studies in this program were limited to laboratory testing, but full evaluation of the use of impregnated aggregates in highway paving systems will require testing under actual field conditions.

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14. Kukacka, L.E., Steinberg, M., and Manowitz, B., Preliminary Cost Estimate for the Radiation-Induced Plastic Impregnation of Concrete, BNL 11263, April 1967.
15. Carter, W.L., "The Paradox of Quarrying in the Northeastern Megalopolis," Environmental Geology, Vol. 1, pp. 67-8, 1975.
16. Grayson, W.J., Jr., "Transportation: The difference between profit and loss," Rock Products, pp. 52-3, June 1977.

APPENDIX A
ANNOTATED BIBLIOGRAPHY

A.1 Impregnants and Impregnation Technology

1. American Concrete Institute, Polymers in Concrete, ACI Committee 548 State of the Art Report, 1977.

"The report describes monomers and polymers used in three types of concrete-polymer composites, the technology for composite production, and the properties of the composite materials. The state of knowledge of engineering design of PIC and PC is summarized, and applications under study or in use for the various concrete-polymer composites, both in the United States and in other countries around the world, are reviewed."

2. Blankenhorn, P.R., et al., "Penetration of Concrete with Various Linseed Oil/Mineral Spirits Mixtures," Cement and Concrete Research, Vol. 9, No. 3, pp. 353-364, 1979.

Reports on laboratory experiments simulating field impregnation techniques.

3. Blankenhorn, P.R., Barnes, D.P., et al., "Residual Porosity After Impregnation of Concrete with Linseed Oil/Mineral Spirits Mixtures," Cement and Concrete Research, Vol. 8, No. 2, pp. 161-168, 1978.

Pore characteristics of concrete impregnation with boiled linseed oil/mineral spirits mixtures were investigated using a mercury porosimeter.

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Summarizes the results of a research program, as of June 1970, investigating the properties of polymer impregnated concrete, as well as potential areas of application.

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Reports on polymer-impregnation of wood, hardened cement paste, mortar, and porous vycor glass.

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A.2 Wear Resistance and Polish Resistance of Aggregates

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4. Burnett, W.C., Gibson, J.L. and Kearney, E.J., "Skid Resistance of Bituminous Surfaces," Research Report 67-3, New York State Department of Transportation, Oct. 1967.

Summarizes the development of a skid trailer and test procedures, and the results of laboratory and field tests.

5. Dahir, S.H., and Henry, J.J., Alternatives for the Optimization of Aggregate and Pavement Properties Related to Friction and Wear Resistance, Final Report, FHWA-RD-78-209, April 1978.

This program was designed (1) to identify and evaluate existing and proposed skid-resistant and wear-resistant aggregate and pavement surfacing systems, and (2) to develop recommendations for material systems that will meet surface performance requirements for skid and wear resistance while taking into consideration other performance properties.

6. Dahir, S.H.M., and Mullen, W.G., "Factors Influencing Aggregate Skid-Resistance Properties," Highway Research Record No. 376, pp. 136-48, 1971.

Reports on factors influencing laboratory skid resistance of paving aggregates, as part of a program on laboratory and field determination of wear and polishing properties of aggregates in relation to pavement skid resistance.

7. Dews, S.J., and Bishop, R.J., "Factors Affecting the Skid-Resistance of Calcined Bauxite," J. Appl. Chem. Biotechnol., pp. 1117-24, 1972.

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8. Fondriest, J.J., and Snyder, M.J., Synthetic Aggregates for Highway Construction, National Cooperative Highway Research Program, Report No. 8, 1964.

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15. Gray, J.E., and Renninger, F.A., "Limestones with Excellent Non-Skid Properties," Crushed Stone Journal, (4), pp. 6-11, 15, Dec. 1960.

The skid resistance of limestone aggregates was analyzed by the insoluble residue test and grain size analysis of the insolubles.

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The polishability of limestone aggregates was evaluated by comparing the polishing characteristics of limestones, as determined by circular test track studies, with mineral and grain size analysis of insoluble residue.

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The petrographic features affecting the degree to which natural aggregates will polish are discussed.

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Results of a national survey conducted to investigate the role of aggregates in slippery pavements.

20. Hosking, J.R., "The Role of Aggregates in Providing Skid - Resistant Roads," Symposium on the Influence of the Road Surface on Skidding, pp. 5.1-5.26. University of Salford, Center for Transport Studies, October 1968.

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25. Kummer, H.W., and White, D.L., Exploratory Field Study of Aggregate - Skid Resistance Effectiveness Report No. 14, Joint Road Friction Program, Penn State University, June 1967.

The effect of aggregate type, shape, and size on skid resistance was determined by measurements with Penn State's skid trailer on 24 test sections placed on a main rural highway. Effects of air and surface temperatures on measured skid numbers are also discussed.

26. McCasland, W., Study of the Wearability and Polish Resistance of Oklahoma's Coarse Aggregates, Final Report, Research and Development Division, Oklahoma Department of Highways, December 1972.

The wearability and polishing characteristics of "non-polishing" aggregate blended with a sound, polish susceptible aggregate were evaluated with the British Accelerated Polish Machine. The effects of crystallinity, grain size, cementing strengths, minerology, and other petrographic features on wearability and polish resistance are also discussed.

27. Moavenzadeh, F., and Goetz, W.H., "Aggregate Degradation in Bituminous Mixtures," Highway Research Record, No. 24, pp. 106-37, 1963.

A laboratory study was done with a gyratory testing machine to determine the factors affecting the degradation of aggregate in bituminous mixtures.

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Laboratory tests were developed for pre-evaluating aggregates and paving mixtures to predict skid resistance in the field and to identify mixtures with optimum skid and polish resistance. Methods include testing on a circular test track machine and petrographic evaluation of polish susceptibility from percentage of hard minerals in the aggregate.

29. Mullen, W.G., "Skid Resistance Laboratory Procedures and Equipment at North Carolina State University," Presented at the Canadian Technical Asphalt Association, Toronto, Canada, Nov. 1976.

Laboratory tests for pre-evaluating skid resistance of aggregates include those using a laboratory scaled circular test track, jar mill method, British pendulum tester, petrographic analysis, insoluble residue test, and variable speed friction tester.

30. Mullen, W.G., Skid Resistance and Wear Properties of Aggregates for Paving Mixtures, Final Report, Project ERSD 110-69-1, North Carolina State University, Sept. 1972.

The program was designed "to develop methods for determining the wear and polishing properties of aggregates in the laboratory and to predict, with reasonable assurance, the limits on polishing of aggregates and mixtures in the field based upon laboratory tests." Laboratory methods developed include use of a small diameter test track, jar mill polishing of loose aggregates, petrographic evaluation of hard mineral content, and insoluble residue test.

31. Mullen, W.G., Whitefield, J.K., Matlock, T.L., and Gibson, D., Implementation For Use of Variable Speed Friction Tester and Small Wheel Circular Track Wear and Polishing Machine for Pavement Skid Resistance, Final Report, Parts I, II and III, ERSD-110-76-2, North Carolina State University, April 1977.

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Laboratory procedures used in England, France, Belgium, and West Germany to evaluate polish and skid resistance of aggregates are reviewed.

33. Patty, T.S., "Petrography As Related to Potential Skid Resistance of Paving Aggregates Used on Texas Highway Pavements," Texas State Department of Highways and Public Transportation, 1975.

Data collected from field observations, laboratory tests with the British Accelerated Polishing Machine, and petrographic analyses are related to the skid resistance of paving aggregates.

34. Patty, T.S., (Texas Highway Department) "Accelerated Polish Test for Coarse Aggregate," Presented at the 24th Annual Highway Geology Symposium, Sheridan, WY, Aug. 1973.

A laboratory method, utilizing the British Accelerated Polishing Machine, is used to evaluate an aggregate's susceptibility to polishing under traffic.

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Summarizes the aggregate properties needed to provide the micro and macro asperities essential in good skid resistant pavement, and tests for pre-determining skid resistance of aggregates.

36. Rose, J.G., and Yavens, J.H., Aggregate Shape and Skid Resistance, Interim Report, KYHPR-71-66, HPR-1(7), Part II, Kentucky Department of Highways, Sept. 1971.

The analysis compares particle angularity and the resultant void percentage with past skid resistance performance.

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Pavement aggregates are polished by a reciprocating wear machine, and polish resistance is measured with the British Pendulum Tester.

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The total acid insoluble residue content of Virginia limestones is related to their polish resistance as evaluated by stopping distance numbers. Tentative guidelines for the use of limestone aggregate in pavement surfaces are based on this relationship and on observations of traffic volumes.

39. Shupe, J.W., and Lounsbury, R.W., "Polishing Characteristics of Mineral Aggregates," Proceedings of the First International Skid Prevention Conference, Part II, pp. 509-37, Virginia Council of Highway Investigation and Research, August 1959.

Reports the polishing characteristics of various mineral aggregates and their relationship to basic aggregate properties, and the effects of texture, as determined by gradation, and of initial aggregate shape on the skid resistance of bituminous mixtures.

40. Smith, N.L., Jr., and Morawski, E.J., Development of a Laboratory Method of Predicting Wear Resistance of Aggregates, Final Report, March 1977.

A small diameter laboratory test track was developed to evaluate the polishability of aggregates.

41. Stapler, W.T., Stone, J.S., and Chase, H.D., A Skid Resistance Study of Georgia Pavements, Final Report, Departmental Research, GHD Research Project No. 6201, Georgia State Highway Department, Dec. 1970.

Reports field test results and the design and operation of a laboratory machine for estimating skid resistance based on field observations and the properties of specimens made in the laboratory. Of primary importance was the effect of the fine and coarse aggregate on the skid resistance of concrete and asphalt pavements.

42. Stephens, J.E., and Goetz, W.H., "Effects of Aggregate Factors on Pavement Friction," HRB Bullentin 302, pp. 1-17, Jan. 1961.

Reports a laboratory study on the effects of aggregate type, shape, and rate of polishing on skid resistance, and tests done to find the optimum surface roughness or texture for a given type of rubber.

43. Stiffler, A.K., "Relation Between Wear and Physical Properties of Roadstones," Highway Research Board Special Report, 101, pp. 56-68, 1968.

The microscopic wear of homogeneous aggregates was studied in the laboratory to determine the physical properties pertinent to the wear process.

44. Stiffler, K., Mineral Wear in Relation to Pavement Slipperiness, Joint Road Friction Program, Report No. 15, Pennsylvania Department of Highways and Penn State University, Nov. 1967.

A laboratory study on a microscopic scale was done on the mechanisms of wear for homogeneous materials occurring separately or as constituents in heterogeneous aggregates.

45. Stutzenberger, W.J., and Havens, J.H., "A Study of the Polishing Characteristics of Limestone and Sandstone Aggregates in Regard to Pavement Slipperiness," HRB Bulletin No. 186, pp. 58-81, Jan. 1958.

Theoretical aspects of pavement slipperiness are discussed, as well as a laboratory study on the fundamental factors affecting frictional skid resistance. A laboratory machine for measuring coefficients of friction for 4-in dia. specimens is described.

46. Tourenq, C., and Fourmaintraux, D., "Properties of Aggregates and Slippery Road Conditions," (in French) Bull Liaison Labs P et Ch, No. 51, pp. 61-9, March-April 1971.

The relationship between the polishing characteristics of aggregates and their mineral composition is investigated.

47. Trollope, D.H., and Wallace, K.B., "The Nature of Skid Resistance," Proc. First Conference Australian Road Research Board, V. 1, pp. 779-92, 1962.

A theory is proposed to explain the reduction in skid resistance at high speeds on wet pavements, and the results of laboratory tests on dry and wet surfaces are presented.

48. Underwood, J.P., Hankins, K.D., and Garana, E., Aggregate Polishing Characteristics: The British Wheel Test and The Insoluble Residue Test, Research Report No. 126-2, Texas Highway Department, 1971.

Aggregate polishing characteristics were evaluated with the British wheel and the insoluble residue test, and the results were compared with field polishing characteristics.

49. Van Til, C.J. et al., Requirements for Wear-Resistant and Skid Resistant Highway Pavement Surfaces, Final Report, NCHRP Project 1-12 (3), March 1976.

Guidelines are presented for the design and construction of ten pavement systems considered most suitable for immediate use where greater than normal skid resistance is required, along with the evaluation of 36 pavement sections under simulated traffic conditions on a circular test track.

50. Walker, D., "The Stone Polishing Test," Symposium on the Influence of the Road Surface on Skidding, pp. 3.1-3.20. University of Salford, Center for Transport Studies, oct. 1968.

Describes the development and operation of the British Accelerated Polishing Machine used to evaluate the tendency of a stone to polish under the action of traffic.

51. West, T.R., and Aughenbaugh, N.B., "The Role of Aggregate Degradation in Highway Construction," Highway Geology Symposium Proc., pp. 117-32, 1964.

Reviews the literature on aggregate degradation, defines the problem, and discusses present and future paths of research.

52. Whitehurst, E.A., "Skid Resistant Pavement Surfaces," Public Works, pp. 73-4, Jan. 1972.

Summarizes current knowledge regarding skid resistance of pavement surfaces and comments on how this can be used to maintain adequate skid resistance of pavements in service.

53. Whitehurst, E.A., and Moore, A.B., "An Evaluation of Several Expanded Aggregates for Use in Skid-Resistant Pavement Surfaces," Journal of Materials, pp. 609-24, Vol. 1, No. 3, Sept. 1966.

The Tennessee Highway Research Program included laboratory and field experiments to evaluate various types of expanded shale aggregates for use in pavement surfacing systems.

54. Woods, K.B. (Editor), Highway Engineering Handbook, 1 ed., McGraw-Hill, 1960.

A compilation of information on highway planning; traffic control; highway administration; finance; economics; materials and design of mixtures; soil mechanics; and design, construction, and maintenance of highways.

55. Woods, K.B., and McLaughlin, J.F., "The Role of Mineral Aggregates in the Design, Construction and Performance of Highway Pavements and Bridges," O.A.S. 9th Pan Am Highway Congress, Document No. 49, Chapter 4, Topic 10, pp. 468-81, May 1963.

Reports on the chemical and physical properties of aggregates as related to the frost resistance of PCC and their potential degradation in base courses and bituminous concrete, on possible adverse chemical reactions of cement and aggregates in PCC, on the influence of aggregates on the skid resistance of pavement surfaces, and on other subjects.

56. Woolf, D.O. and Runner, D.G., "The Los Angeles Abrasion Machine for Determining the Quality of Coarse Aggregate," ASTM Proc, Vol. 35, Part II, pp. 511-32, 1935.

A test method for coarse aggregate using the Los Angeles abrasion machine is discussed and field service behavior is compared with the losses in the Deval and Los Angeles abrasion tests.

APPENDIX B
PETROGRAPHIC ANALYSES OF TEST AGGREGATES

Aggregate Code No:	0901 (Figure 16)
Rock Type:	Limestone
Source:	E.R. Jahna Industries Lacanto Hard Rock Plant Pit Number 02-35 Lacanto, Florida

<u>Constituent Minerals</u>	<u>Volume, %</u>	<u>Grain Size (mm)</u>
Calcite	80	Micrite, <0.01 (80%) Spar, ~ 0.02 (20%)
Opagues	tr	
Pore Space	20	

Macroscopically the rock is a white, porous limestone. The bulk of the rock consists of round (in cross section) and ovoid polycrystalline particles, typically 0.5 mm in diameter, composed of submicroscopic (<0.01 mm) calcite grains. Lack of concentric zoning in most particles suggests detrital fecal pellets. Concentric zoning in some indicates a small proportion of oolites. Fossils and fragments of fossils are also present. Fossils consisting of single calcite grains up to 0.5 mm in diameter are rare. The detrital material (pellets, oolites, and fossils) is only partially cemented together by anhedral calcite grains averaging 0.02 mm in diameter. Much of the pore space is unfilled.

Aggregate Code No:	2307 (Figure 17)
Rock Type:	Dolomite
Source:	Brian Aggregate Co. Shakopee, Minnesota

<u>Constituent Minerals</u>	<u>Volume, %</u>	<u>Grain Size (mm)</u>
Dolomite	80	~0.2
Quartz	5	0.02
Opakes	tr	
Pore Space	15	

The rock is a homogeneous, reddish-brown, porous dolomite. The texture and mineralogy are uniform throughout the section. Euhedral dolomite rhombs are partially cemented by concentrically layered overgrowths. Cores are cloudy, with minute reddish-brown opaque inclusions. Rims are clear and in optical continuity with the cores. Interstices between rhombs are not entirely filled, and there is pore space. Many rhombs show partial to complete replacement of their cores by fine grained, polycrystalline aggregates of quartz. The replacement of cores but not overgrowths suggests that quartz replacement occurred prior to formation of the overgrowths.

Aggregate Code No:	4315 (Figure 18)
Rock Type:	Volcanic Lithic Arenite (Sandstone)
Source:	Leo P. Cloud and Son SanStone Materials-Pawelekville Pit San Antonio, Texas

<u>Constituent Minerals</u>	<u>Volume, %</u>	<u>Grain Size (mm)</u>
Lithic Fragments	35	~0.4
Quartz	30	~0.4
Plagioclase	15	
Matrix	15	
Alkali Feldspar	3	
Muscovite	1	
Tourmaline	1	

Arenite indicates that it is composed of dominantly sand sized grains; the term lithic indicates that a significant proportion of these grains are fragments of other rocks rather than individual minerals; and the term volcanic indicates that most of the detritus is probably volcanic in origin. This is indicated by the presence of fine grained glass, well developed zoning in some plagioclase, embayments on some quartz and plagioclase pumice, spherulitic felsic material, and fragments of nearly aphanitic volcanic rocks. These characteristics, together with the textural immaturity and the mineralogy, indicate that this rock represents a first-cycle reworking of material derived from a high SiO₂ volcanic terrain.

Aggregate Code No:

4801 (Figure 19)

Rock Type:

Quartzite

Source:

Tony Pacifico Quarry
Charleston, West Virginia

<u>Constituent Minerals</u>	<u>Volume, %</u>	<u>Grain Size (mm)</u>
Quartz	80	0.1-2.0
Plagioclase	tr	
Alkali Feldspar	tr	
Muscovite	tr	
Biotite	tr	
Chlorite	tr	
Opakes	tr	
Pore Space	20	

The rock is a moderately well-sorted porous quartzite. Detrital grains are mostly rounded to angular single crystals of quartz with minor amounts of feldspars, muscovite, biotite, and chlorite. Quartz grains are commonly strained, some showing advanced stages of polygonization. Optically continuous overgrowths are present on many quartz grains. Alkali feldspars are highly altered to sericite. Grain boundaries are mostly tangential or point contacts, though sutured contacts are not uncommon. The porosity is high but difficult to estimate accurately because grains were plucked during cutting and polishing of the thin section. Individual grains are extremely hard, but they are only loosely bound together.

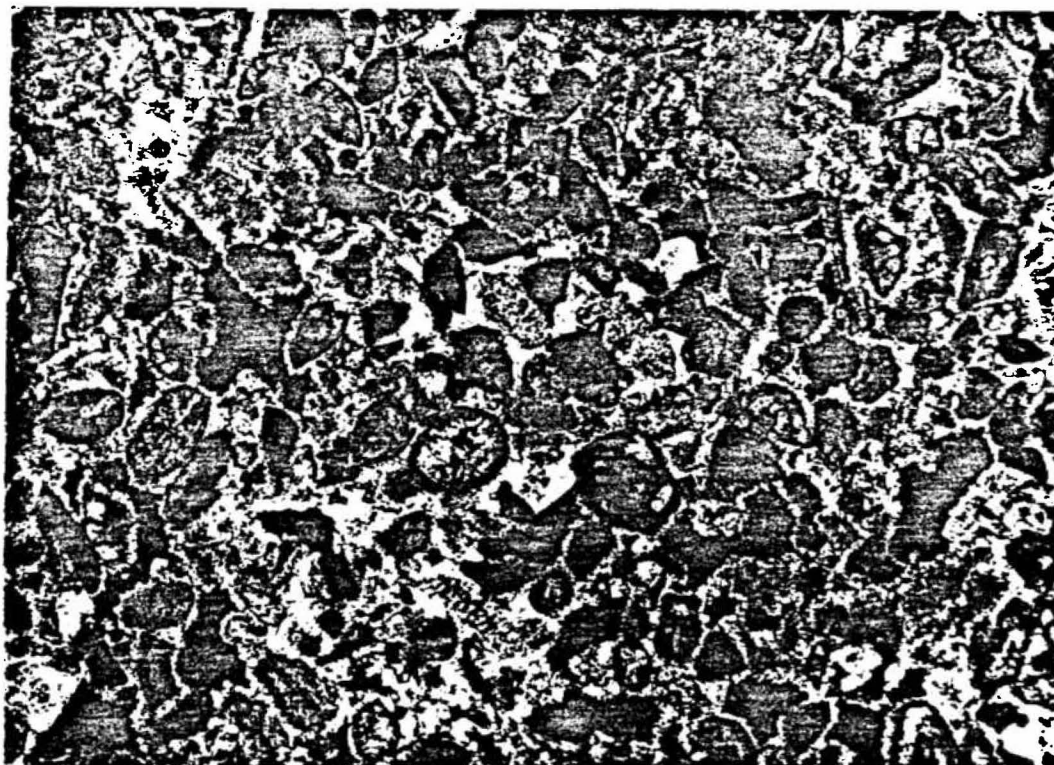


Figure 16 (X40) Aggregate 0901, limestone (x-polars).



Figure 17 (X40) Aggregate 2307, dolomite (x-polars).

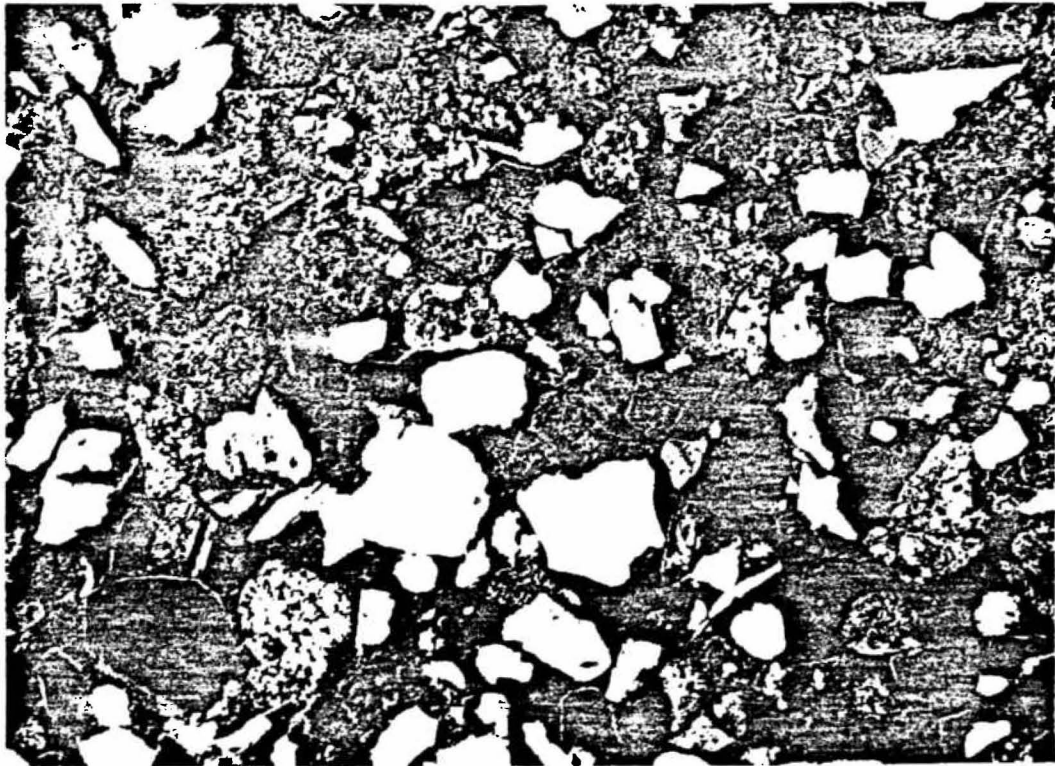


Figure 18. (X40) Aggregate 4315, lithic arenite (x-polars).



Figure 19 (X-40) Aggregate 4801, quartzite (x-polars).

APPENDIX C
SELECTED IMPREGNANTS AND LABORATORY IMPREGNATION PROCEDURE

The five impregnants selected for evaluation are described, and the laboratory impregnation process is discussed.

C.1 Selected Impregnants

(1) 90 wt% methyl methacrylate (MMA) - 10 wt% trimethylolpropane trimethacrylate (TMPTMA). This low viscosity monomer system, ~1 mPa·s (1 cP) at 24°C (75°F), was used extensively in the early research on polymer impregnated concrete. MMA is a low viscosity monomer, ~0.85 mPa·s (0.85 cP) at 24°C (75°F), which produces a high strength polymer with excellent durability. TMPTMA is a comonomer that is added to the system to improve the strength and durability by serving as a cross-linking agent. The polymerization is thermal-catalytic, with 1 wt% initiator (2-t-butylazo-2-cyanopropane) added to the monomer system prior to impregnation. After impregnation, the aggregate is heated to a minimum temperature of 80°C (176°F). The heat breaks down the initiator into free radicals, which generate the polymerization reaction.

(2) 83 wt% MMA - 5 wt% TMPTMA - 12 wt% poly(methyl methacrylate) (PMMA). This relatively low viscosity monomer system, ~15 mPa·s (15 cP) at 24°C (75°F), produces a polymer having good strength and durability. A medium molecular weight PMMA is used to increase the viscosity of the monomer and thus reduce monomer damage and evaporation loss in the aggregate prior to polymerization; however, it also lowers the thermal stability of the resultant polymer. One percent silane coupling agent (γ -methacryloxypropyltrimethoxysilane) is added to the monomer system to improve the bond between the polymer and the aggregate by developing a chemical bond between the polymer and any silica present in the aggregate. The monomer system is polymerized thermal-catalytically by adding 1 wt% initiator (2-t-butylazo-2-cyanopropane) and heating to a minimum temperature of 80°C (176°F).

(3) 57 wt% styrene - 38 wt% TMPTMA - 5 wt% polystyrene. This is a relatively low viscosity monomer system, ~15 mPa·s (15 cP) at 24°C (75°F), which produces a strong polymer with good durability and better thermal properties than the MMA-based systems. The styrene monomer serves as the base for the system. The TMPTMA is added to serve as a cross-linking agent, to improve strength and durability. The polystyrene is added to increase the viscosity of the system and thus reduce monomer damage and evaporation loss prior to impregnation. One percent silane coupling agent (γ -methacryloxypropyltrimethoxysilane) is added to improve the bond between the polymer and the aggregate. Polymerization is done thermal-catalytically with 1 wt% initiator (2-t-butylazo-2-cyanopropane) and heating to a minimum temperature of 80°C (176°F).

(4) Phenol Formaldehyde Resin (Monsanto RESINOX RS-7288). This is a water dilutable, general purpose phenolic resin. The viscosity is quite high, ~190 mPa·s (190 cP) at 24°C (75°F) but can be reduced as low as 47 mPa·s (47 cP) with the addition of water. The resin produces a polymer with good strength and durability and excellent thermal stability. It is fully cured by heating to 138° to 177°C (280° to 350°F), essentially driving off the water.

(5) Sodium Silicate Solution. This is a water based solution consisting of 37% sodium silicate ($1 \text{ Na}_2\text{O} \cdot 2.8 \text{ SiO}_2$). Its initial viscosity of ~257 mPa·s (257 cP) at 24°C (75°F) can be reduced by adding water. The solution is cured by driving off the water with heat, producing an extremely strong and durable adhesive.

C.2 Laboratory Impregnation Process

The process used in the laboratory to impregnate the aggregates was as follows. The aggregate was placed on a screen and washed with running water to remove any dirt, clay, or other deleterious materials adhering to the surface, and then dried in an oven at 150°C (302°F) to constant weight and then allowed to cool to room temperature, 24°C (75°F). The aggregate was put in a wire basket (Figure 20) and placed inside the impregnation vessel (Figure 21). The vessel was sealed and evacuated to 760 mm Hg for a minimum of 60 min. Then the impregnant was introduced into the sealed vessel, and an overpressure of 103 kPa (15 psig) was applied for 4 hr with nitrogen. After this, the excess impregnant was drained from the vessel, and the impregnant within the aggregate was cured. The curing method varied, depending upon the impregnant being used.

The MMA-based systems and the styrene-based systems were cured by injecting steam, at approximately 112°C (234°F), into the impregnation vessel for a minimum of 2 hr after complete draining of the excess impregnant. The phenol formaldehyde and the sodium silicate impregnated aggregates were cured by placing them on screens or in flat pans in an oven for 12 hr to drive off the water, the former at 150°C (302°F) and latter at 115°C (234°F).

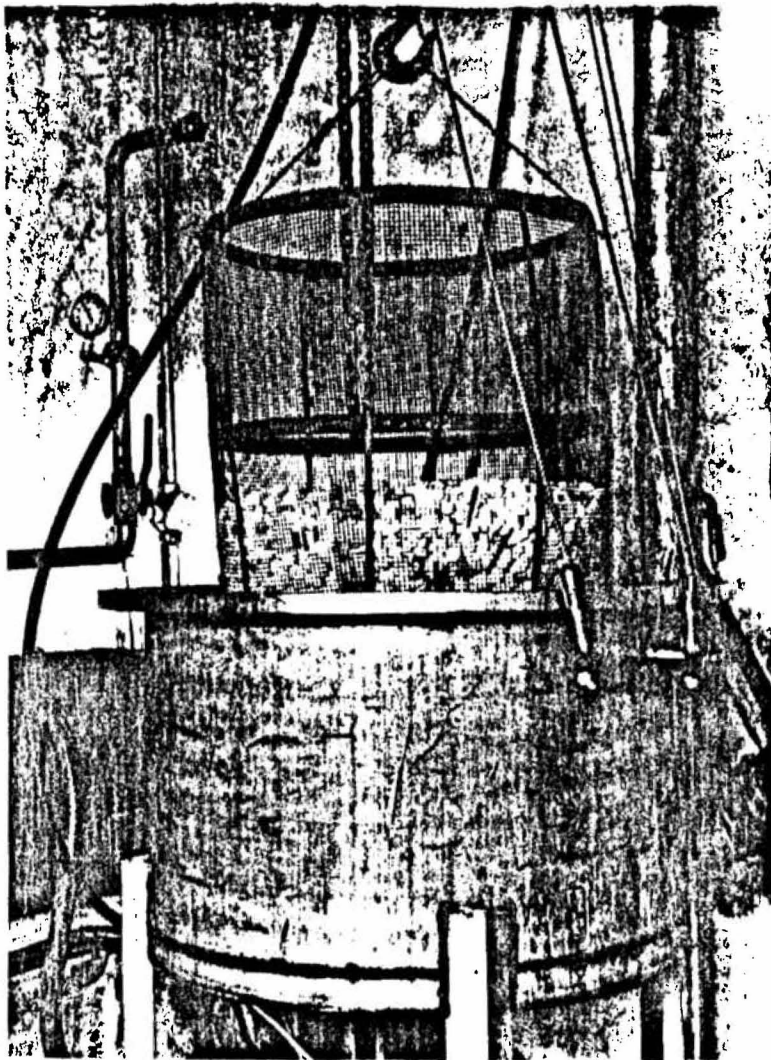


Figure 20 Placement of Aggregate in Impregnation Vessel.

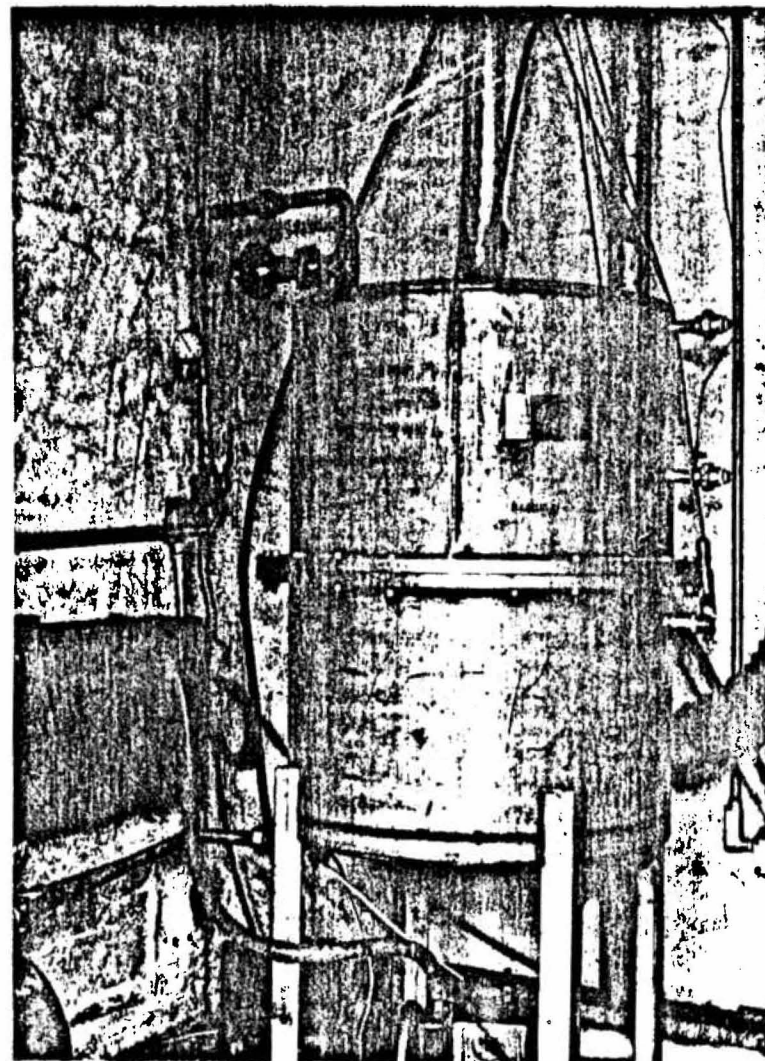


Figure 21 Sealed Impregnation Vessel.

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