Porous Asphalt Durability Test

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Contents

Executive Summary

The most common form of distress of aged open graded porous asphalt (OGPA) is loss of chip from the surface (fretting and ravelling) caused by embrittlement through reaction with atmospheric oxygen. Resistance to chip loss (the durability of the mix) depends not only on the oxidation resistance of the binder but also on binder film thickness, aggregate grading and percentage of air voids. At present there exists no satisfactory test to assess the effect of oxidation on compacted OGPA. This research aimed to develop a test to compare the effect of different binders and mix design parameters on OGPA durability.

A review of the literature revealed relatively little work had been carried out in relation to oxidation and performance assessment of compacted mix in general, and even less on OGPA in particular. Most researchers have used low temperatures to prevent binder drainage and disruption of the mix, and measured abrasion resistance with the cantabro test. The conditions used in the cantabro test vary somewhat, but typically involve revolving a compacted, Marshall test sized cylinder of mix in a steel drum, for 300 revolutions at 30 rpm and at temperatures up to 25°C. The weight lost from the specimen is recorded as a percentage of the original weight. An important consideration for test development was the relationship between the effect of the oxidation procedure used and field ageing. Most studies have investigated effects such as binder type on oxidation resistance without specifically addressing field ageing. For dense mixes (<10% voids), the US Strategic Highway research programme developed standard short term (to model plant hardening), and long term ageing procedures (to model field hardening). In the short term test loose mix is oxidised at 135°C for four hours and then made up into Marshall sized specimens for the long term test, which comprises a further five days' heating at 85°C. These tests were found to represent 5-15 years of field ageing in the US, depending on climate. However Khalid (2002) found that for OGPA the tests appeared to be equivalent to only a few months in the field in the UK. Clearly numerous variables are involved in determining oxidation rates and the need to obtain local data for comparison to laboratory results is highlighted.

In the present study the bitumen from field samples of OGPA of various ages was extracted and the viscosity at 60°C measured. Comparison of the field data with results from preliminary experiments on compacted OGPA indicated that oxidation at temperatures up to 85°C was too slow for practical purposes. As higher temperatures would increase the risk of damage to the specimens, oxidation was accelerated by increasing the air pressure to 2070 kPa (300 psi). Three days oxidation at 80°C and 2070 kPa air pressure was found to increase binder viscosity to a level approximately equivalent to 4.5 years in the field and these conditions were adopted. The abrasion resistance of the oxidised mix was measured and compared to that of the unaged mix using the cantabro test (300 revolutions at 30 rpm and at 25°C).

The possibility that the use of high air pressures in the test may mechanically damage the compacted specimens was investigated. Although no visible damage occurred,

specimen dimensions increased slightly. To find out if this was significant specimens were tested using an inert nitrogen atmosphere at 2070 kPa to preclude oxidation, leaving other variables unchanged. Cantabro test results showed no significant difference from untreated specimens.

The ageing procedure was applied to laboratory-prepared OGPA specimens and to samples of TNZ class PA14 and PA14HS mixes obtained from an asphalt plant during normal operation. All mixes met the requirements of the Transit New Zealand P/11 specification (TNZ 2003). Relative to un-aged specimens, cantabro test weight losses for oxidised mix increased by factors of 2.1, 1.9 and 1.5 for the laboratory prepared and the plant PA14 and PA14HS mixes respectively. In absolute terms the oxidised laboratory mix had the highest loss (25.9%), while the plant mixes lost 14.0% (PA14) and 13.3%(PA14HS) of their weight.

On this basis a limit for acceptable weight loss after the test could be set at 30%. Based on the three materials studied an appropriate design criterion for cantabro losses of the un-oxidised mixes would be 15%. These tentative figures should be confirmed by testing additional mixes.

Abstract

A simple laboratory test procedure to accelerate the oxidation of compacted open graded asphalt mix specimens has been developed. It involves heating 100mm dia x 65mm high cylinders of asphalt at 80°C for 3 days under air pressure of 2070 kPa (300psi). Comparison of the viscosity of the recovered binder with that from field samples of aged OGPA shows that the procedure results in oxidation equivalent to that from about 4.5 years in the field. The effect of binder oxidation on the abrasion resistance of the mix is measured using the cantabro test (300 revolutions at 30 rpm, 25°C).

The test was applied to laboratory prepared OGPA specimens and to samples of two different mixes (PA14 and PA 14HS) obtained from an asphalt plant during normal operation. All mixes met the requirements of the Transit New Zealand P/11 specification. Compared with untreated specimens, cantabro weight losses were greater by factors of 2.1, 1.9 and 1.5 for laboratory prepared, plant PA14 and plant PA14HS mixes respectively. The highest average mass loss of 25.9% occurred with the laboratory mix. For the plant PA14 and PA 14HS mixes, losses were 4.0% and 13.3% respectively. Measurement of changes in specimen dimensions after the test, and cantabro test results from experiments using 2070 kPa of nitrogen instead of air, show that the high pressures used do not significantly affect the integrity of the specimens.

1. Introduction

The aim of this research was to develop a laboratory-based test procedure to model field durability (oxidation) of compacted porous asphalt, allowing comparative assessments of the likely cohesion and resistance to fretting of OGPA after some years in the field.

Such a test procedure is needed to compare the durability of different porous asphalt mix designs and binder types, for example polymer modified binders and unmodified 60/70 or 80/100 penetration bitumens. It is hoped that the procedure will provide a platform for the development of durability criteria to assess the long-term field performance of open graded mixes in the Transit New Zealand performance-based specification, P/23, currently being formulated.

The safety and environmental advantages of porous asphalt compared with other surfacings are well known and include:

- Improved wet weather skid resistance and reduced risk of aquaplaning.
- Less splash and spray in wet weather.
- Reduced reflection from wet surfaces giving better visibility of road markings.
- Reduced noise inside and outside the vehicle.

For example a study in the UK (Nicholls 1997) found that compared with rolled asphalt of equivalent texture, noise from porous asphalt was 3-5dBa less depending on its age. Spray was reduced by 95% initially and even after 8 years by 66%.

Two major problems with porous asphalt are clogging of the voids and oxidation of the binder.

Clogging of the voids simply causes a gradual fall in noise and spray reduction performance to lower, but still acceptable, levels closer to that of conventional dense graded mix. Binder oxidation, however, is the main factor limiting the life of porous asphalt, whose very open nature allows faster oxidation and binder embrittlement than in conventional mixes. This leads ultimately to failure of the mix through loss of material from the surface (ravelling or fretting) under traffic shearing stresses, making it rough and uneven. The short average lifetime (Bartley Consultants 1999) in New Zealand of 10.5 years for porous asphalts (in many cases only 7-8 years), compared with that of dense mixes (16.2 years), adversely affects their benefit-cost ratios, inhibiting widespread use of this safe and environmentally friendly surfacing.

In an attempt to improve lifetimes, many roading engineers use expensive polymer-modified binders (PMBs) in porous asphalt mix. To properly evaluate the benefit of these and other modifiers such as fibres, a procedure is needed to predict likely fretting and cohesion of mixes after several years in the field. The current Transit New Zealand P/11 specification (TNZ 2003) does not directly address long-term resistance to fretting caused by oxidation.

2. Development of an OGPA durability test

2.1 Test design

The most common form of distress of aged OGPA is loss of chip from the surface (fretting and ravelling), caused by embrittlement of the bitumen after reaction with atmospheric oxygen. This form of distress is believed to involve aggregate loss through cohesive failure of the bitumen or possibly failure at the bitumen/aggregate interface. In the latter case such adhesion failure should be distinguished from water-induced stripping, which tends to occur only when the mix is kept saturated with water. A primary role for water stripping in OGPA surface fretting seems unlikely.

One way to assess durability would be to measure the oxidation behaviour of the bitumen alone and try to identify a level of viscosity (for example) at which fretting begins. However, this would ignore factors such as void content and inter-connectedness, binder film thickness and aggregate gradation, all of which affect the rate of bitumen oxidation and the ability of the aged mix to withstand traffic stress. Therefore a test to compare the durability of different OGPA formulations needs ideally to be based around the ageing and the resulting abrasion resistance properties of the compacted mix, rather than predicted solely from the behaviour of the oxidised binder.

A suitable test method must include procedures to (a) accelerate the oxidation of compacted mix specimens and (b) measure the susceptibility of mixes to oxidation in terms of abrasion resistance (as fretting is the major failure mode in the field).

It was decided from the outset of this work to measure abrasion resistance using the cantabro test (see Appendix A.2), in which weighed blocks of mix are tumbled in a steel drum and the percentage weight of aggregate lost is recorded. Variations of the test are widely used internationally, and it is increasingly being used in New Zealand to assess the cohesion of OGPA at construction. At the time of writing the cantabro test is likely to be incorporated into the performance based P/23 OGPA specification under development by Land Transport New Zealand. Because a durability test may also form part of that specification, it was thought logical to use cantabro equipment in this work.

The cantabro test procedure used was that given in APRG 18 (APRG 1999). A criticism of the test is that it the way in which it stresses the specimens (by rotating in a drum) appears to bear little relation to stress caused by traffic. It is thus probably impossible to extrapolate and predict field performance in an absolute sense with any confidence. However, for comparative measurements the cantabro test is simple, inexpensive and quick. The method in APRG 18 specifies a test temperature of 25°C, although research indicates that the use of temperatures below 0°C may provide greater differentiation between binder types (Perez-Jimenez et al 1999). The European specification for the test specifies a temperature of 10°C (BS EN 12697-17 2004). Such low temperatures are expensive to maintain, so to facilitate practical implementation a temperature of 25°C was adopted for this work..

Methods used to accelerate the oxidation of compacted mix must do so without significant binder run-off or disruption to the structure of the mix. The degree of oxidation achieved in the laboratory must be able to be compared with field oxidation. Therefore, field samples of OGPA surfacings of various ages were collected, analysed and the data compared with laboratory aged samples (section 2.3).

2.2 Oxidation of compacted asphalt mixes- literature survey

Because the oxidation of bituminous paving mixtures is of considerable practical importance, much effort has been made in developing test methods to assess and compare long term in-service durability. Airey (2003) and Bell (1989) reviewed bitumen oxidation research from an engineering perspective. Most of the research reviewed has focussed on methods to accelerate and assess the effect of oxidation of the bitumen alone or of loose mix. Less attention has been paid to methods to accelerate the oxidation of compacted mix and to evaluate its durability.

2.2.1 Dense graded mixes

Lang and Thomas (1939) oxidised Ottawa sand briquettes using heat and in some cases ultraviolet light. They carried out abrasion tests on the aged mixes, extracted the binders and measured their properties. Details of their findings are not readily available.

Pauls and Welborn (1952) cured a mix of Ottawa sand as a thin layer for 0.5 hour at 163°C, then compacted it into 50.1 mm high x 50.1 mm diameter cylinders which they heated at 163°C for up to 8 hours. They measured the compressive strength of the cylinders, recovered the binder and measured the penetration, ductility and softening point. They did not correlate the laboratory results to field data.

In a study to examine the effect of lime on bitumen oxidation, Plancher et al (1976) oxidised 25 mm high x 40 mm diameter cylinders of mix at 150°C for 5 hours and compared the resilient moduli at 25°C with those of untreated specimens. They found that lime reduced the rate of viscosity increase, and discussed possible mechanisms.

Hveem et al (1963) aged ‘semi-compacted’ Ottawa sand mixtures at 60°C using infra-red heating in an air stream at 41°C. They carried out shot abrasion tests on the oxidised and compacted specimens and compared the results with field data from conventional asphalt mix. 1000 hours of the ageing treatment was found to be approximately equivalent to five years’ field ageing in California.

Goode and Lufsey (1965) studied the effect of air voids (up to 10%), bitumen film thickness and permeability on the oxidation of 63.5 mm high x 101.1 mm diameter compacted asphalt specimens. They aged the specimens at 60°C in an oven for up to 63 days and for 303 days outside, then recorded the Marshall properties of the mix specimens and the penetration and softening point of the extracted binders. The data in their tables VIIA and VIIIA show a significantly greater increase in Marshall stability after 303 days in the field than after 12 days’ laboratory ageing. Mixes aged for 63 days produced results roughly comparable to those from 303 days of field ageing.

Kumar and Goetz (1977) studied the effect of air voids (up to 16%), bitumen film thickness and permeability on the oxidation of compacted Marshall specimens. They oxidised the mix for up to 18 days at 60°C by drawing a stream of air through it using a 0.5 mm Hg vacuum, then measured the effect by creep test.

Hugo and Kennedy (1985) compacted asphalt mix into slabs from which they cut 40 mm high x 100 mm diameter cores. They heated these at 100°C for up to 7 days (in some cases at above 80% relative humidity), and treated some of them with ultraviolet light and in a weatherometer. They then extracted the bitumen and measured its viscosity. Factors such as the surface texture and air voids of the mix affected relative oxidation behaviour.

Kim et al (1986) used pure oxygen at 690 kPa (100 psi) and 60°C to oxidise 4-12% air void Marshall specimens for five days. They measured the effect of oxidation by changes in the resilient moduli and fatigue life, and investigated factors such as compaction level. Based on compositional analyses of bitumen samples oxidised under these conditions, they estimated that the procedure resulted in 5-10 years' equivalent field ageing.

Tia et al (1988) also used indirect tensile strength and resilient modulus to evaluate ageing. They oxidised compacted mix at 60°C (with and without ultraviolet light exposure) for up to 90 days; and also measured properties, such as penetration and viscosity at 60°C, of the extracted binders.

Bell and co-workers at the University of Oregon extensively investigated oxidation of compacted mix under the United States SHRP research programme of the early 1990's (AbWahab et al 1993, Bell 1989, Bell et al 1992a, Bell et al 1992b, Bell et al 1994a, 1994b, 1994c, 1994d, SHRP 1994a, SHRP 1994b, Sosnovske et al 1993). Their work drew in part on the earlier study of Von Quintas 1988. In summary, a short term oven ageing (STOA) method was developed to simulated hardening during manufacture and handling, while a long term (LTOA) method replicated oxidation in the field. Detailed descriptions of the procedures are in Harrigan et al 1994. Under the STOA method loose mix is heated in trays (spread at about 20-22kg/m^2) at 135°C for four hours with hourly stirring. The LTOA method involved compacting the STOA mix and modelled long term ageing by heating the specimens at 85°C for five days. Measurements on the aged specimens included resilient modulus, indirect tensile strength and dynamic mechanical analyses. The researchers also investigated the use of higher temperatures (100°C or 107°C) and high air or oxygen pressures (up to 2070 kPa or 300psi) on dense mixes with air voids of 4-10%. However, these conditions disrupted the mix and were not recommended for dense mix. The SHRP researchers also investigated a low pressure 345 kPa (50 psi) method in which they passed oxygen or air through a specimen at 85°C for five days in a modified triaxial test cell. They recommended this method for open graded mixes, but presented no data for high void mixes. Correlation with field sites (typically 5% air voids) showed that the STOA method was roughly equivalent to 0-2 years in the field, and the LTOA method to 5-15 years, depending on climate.

Kandhal and Chakraborty (1996) used the SHRP STOA and LTOA methods to study the effect of film thickness on ageing behaviour measured in terms of resilient moduli, tensile properties and recovered binder properties.

Brown and Scholz (1996) used protocols similar to those of the SHRP research to investigate the ageing of asphalt mixes. They heated loose mix in a tray for various times at 135°C, compacted it (to around 3% air voids) and measured the stiffness modulus. They found that about 2 hours at 135°C was representative of ageing during plant manufacture and handling. Long-term oxidation was carried out at 85°C for various times but field data were not available for comparison.

Korsgaard et al. (1996) oxidised compacted mix specimens (5% bitumen, 1.1-5.8% air voids) using the pressure ageing vessel (PAV) test developed under the SHRP research programme for oxidising bitumen. They oxidised the mix specimens at 100°C and 2.1 MPa of air for up to 72 hours. The penetration and softening point of the extracted bitumen were measured. They reported that the rate of oxidation of pure bitumen (presumably as 3.2 mm films) was 4 times faster than that of bitumen in the mix.

Hachiya et al (2003) prepared compacted slabs of dense mix and cut them into 20x40x250 mm beams. They oxidised these in an oven at 70°C for up to eight hours and measured the flexural strength, strain at failure and stiffness of the mix at –10° to 30°C. They also carried out oxidation experiments at 60°C in a pure oxygen atmosphere for up to 20 days. Solvent and chromatographic separations and penetrations of extracted bitumen were also measured. The laboratory results were compared with data from samples exposed outdoors for up to five years, showing similar trends (though with quantitative differences); and the authors conclude that a combination of the two oxidation methods would be most suitable. Their data seem to show that measured flexural properties were relatively insensitive to oxidation time (as opposed to test temperature). Though the authors do not correlate laboratory oxidation time to field ageing period, their data suggest that five days’ exposure to pure oxygen at 60°C is approximately equivalent to 3 years’ field ageing, based on flexural properties over 0°C.

Collop et al. (2004) used high pressure air (2.1 MPa) at 85°C in the presence of water to oxidise compacted mix specimens (4% bitumen, 8% air voids). The object of their work was to develop a method to simulate suspected water damage occurring in high modulus base asphalt in the UK. They measured mix oxidation in terms of the indirect tensile stiffness modulus (ITSM). They found that when water saturated mix was oxidised (with excess water present) the ITSM decrease was significantly greater than that of oxidation under the same conditions but with dry specimens. Oxidation by heating in an oven at 85°C, or at 85°C with 3 L/min air flowing through the specimen (the method of Khalid 2002) markedly increased the ITSM compared to the control. The implication to be drawn from their work is that high air pressures are damaging the structure of the mix and that this effect is accentuated by water.

2.2.2 Open graded mixes

The work discussed above dealt almost exclusively with ageing of dense mixes. Fewer reports have specifically discussed the ageing of open graded mixes.

Vonk et al (1993) stored blocks of open graded mix in an oven at 50°C for periods of up to 36 weeks. They measured the abrasion resistance of the mix by the California fretting test (similar to the cantabro test but with the addition of steel ball bearings) and tested the extracted binders. They found that 5-7% SBS polymer significantly improved fretting resistance. They did not relate their results to an equivalent field age.

Mallick et al (2000) aged Marshall specimens (12.5-15.1 % air voids) at 60°C for 168 hours (seven days) in an oven. They measured abrasion loss by the Cantabro test (see appendix A.2). For four different mix designs, mass loss increased by 1.5 to 2.1 times compared with untreated specimens. The researchers set a mix design criterion that the average loss from five aged specimens should not exceed 30% (and no individual result should exceed 50%) compared with an upper limit of 20% for the average of un-aged specimens. They did not relate their results to an equivalent field age.

Miro and Perez-Jimenez (2001) and Perez-Jimenez et al (1999) used the cantabro test at 25°C to assess the effect of oxidation at 163°C for eight hours on compacted, open graded (27-29% air voids) mix Marshall specimens. They confined the specimens laterally with a wire mesh and held them in a frame that allowed turning every two hours to minimise binder runoff. Based on the penetration of extracted binder, five hours' testing gave ageing equivalent to more than five years in the field. Results showed the durability of the mix to depend on binder type, film thickness and air voids. However, previous research shows that bitumen from different sources can have different temperature-oxidation rate dependencies (Branthaver et al 1993), so the very high test temperature used may be questionable for modelling field ageing in general. In an earlier study (1999) Perez-Jimenez et al aged specimens at 80°C for up to 90 days and did cantabro tests on them at temperatures down to -30°C. The results depended on binder type and penetration. Cantabro test results at temperatures below 10°C showed more differentiation between binders than those at 25°C.

Khalid (2002) used an air flow of 3L/min to oxidise Marshall specimens of porous asphalt at 60°C for periods of up to 21 days. The bitumen content was 4.5% but air voids were not reported. They sealed the sides of the specimens to ensure the air passed through the centre of the mix. Oxidation was measured by changes in the indirect tensile modulus and by the complex moduli of the recovered binders. The results were compared with those for specimens subjected to the SHRP STOA (4 hours at 135°C) and LTOA (5 days at 85°C) procedures (see section 2.2.1). For unmodified 100 penetration bitumen the test exceeded the severity of the LTOA test after four days. Comparison with specimens exposed for 18 months on a rooftop in the UK (in air temperatures from 29.7 to –3.6°C) showed that even after 21 days' ageing at 60°C, the measured changes in moduli ratios were less severe than those for the rooftop specimens. The STOA and LTOA procedures appeared to represent less than three and six months' outdoor exposure respectively.

Clifford et al (1996) also used the SHRP STOA and LTOA procedures and subjected the resulting oxidised blocks to the cantabro test (presumably at 25°C). They did not relate their results to field ageing.

Tolman and van Gorkum (1996) aged OGPA blocks by holding them successively in air (50°C for 16.25 hours), salt water (40°C, 4 hours), fresh water (20°C, 1 hour) and cold air (-20°C, 2.75 hours). They then subjected the blocks to a dynamic direct tensile test and plotted the deformation as a function of the number of loading cycles. Their oxidation procedure is relatively mild in comparison with others reviewed. They state they oxidised their samples to the equivalent of up to 9 years in the field, but give no details.

Nielsen et al (2004), as part of a study on porous asphalt durability, used the SHRP LTOA procedure to oxidise a range of mixes of various designs and with different binder additives (at about 20% air voids). They compared the effect of oxidation on the mixes using the cantabro test at 10°C, and measured softening points and penetrations on the extracted binders.

2.3 OGPA oxidation in the field in New Zealand

A detailed study of OGPA oxidation in the field would need to take into account the effect of initial air voids, binder content, climate, variation in penetration within the 80/100 and 60/70 envelopes and the source of crude oil from which the bitumen was derived. For most sites in this study (see Table 2.1), especially older ones, these details were not readily available. Instead, the study determined typical bitumen properties after various periods in the field, and compared them with laboratory oxidised samples.

OGPA sites of various ages, in which the mix was manufactured using only 80/100 penetration grade bitumen, were identified and cored for analysis. The mix at most sites had been manufactured according to TNZ P/11, but that at some older sites is likely to have been the earlier ‘Friction Course’ type. Details are in Table 2.1.

Table 2.1 OGPA field sites.

Site	Location	Age (years)
1	Lower Hutt, Wainuiomata Road	13.0
2	Lower Hutt, Hutt Road	7.7
3	Lower Hutt, Normandale Road	0.4
4	New Plymouth, Smart Road	4.3
5	Wellington, State Highway 2	20.0
6	Invercargill, Tay Street	5.0
7	Invercargill, State Highway 6	8.0
8	Porirua, Cobham Drive	4.1
9	Upper Hutt, River Road	5.6

Cores were taken from the shoulders of lanes to avoid artefacts from oil contamination. The cores were too thin to allow direct measurement of their properties (eg moduli, cantabro test) by standard methods. Instead, to determine the extent of oxidation of the bitumen in the mix, it was extracted with the solvent dichloromethane. Because solvent extraction may irreversibly alter the physical distribution of the polymer in the bitumen and affect the resulting properties of the binder, polymer-modified OGPAs were excluded.

The bitumen in the cores was extracted and recovered as described in Appendix A.1. Figure 2.1 shows the effect of age on the viscosity at 60°C. The zero time point was obtained by extracting a newly produced plant mix sample manufactured with an 80/100 bitumen. Given the number of variables affecting oxidation, the results show a considerable scatter, as expected. The point labelled ‘a’ represents a four year old OGPA that was already severely cracked and scheduled for replacement, and may be anomalous. Overheating or other manufacturing problems may have contributed to the high viscosity of this sample. All sites over 6 years old exhibited surface fretting to varying degrees. Data from the 13 and 20-year old sites (‘b’ and ‘c’ respectively) appear to show a marked lessening in rate of viscosity increase with time. Similar results have been observed in previous studies on field ageing of dense mix (for example Bell 1989 and references therein), but extra sites are needed to confirm these results. A best fit line was drawn excluding points a, b and c.

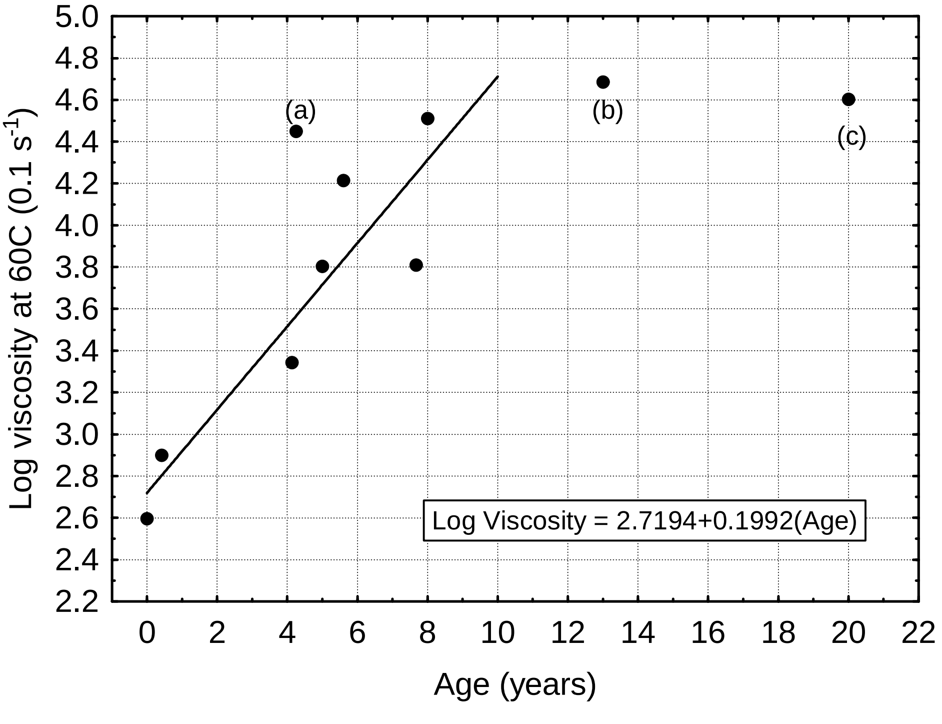

Figure 2.1 Recovered bitumen viscosities from OGPA field sites.

2.4 Laboratory test development

2.4.1 Mix design

Blocks of OGPA (meeting the requirements of TNZ P/11 PA 14 type) were prepared according to Appendix A.2 using 80/100grade bitumen. Bitumen content was 4.7%, and the mix aggregate grading is given in Table 2.2. Average air voids were 19.1 ± 0.8 % (95% confidence limits).

Table 2.2 OGPA grading curve

Sieve Size	13.20	9.50	6.70	4.75	2.36	0.60	0.075
% Passing	100	98	66	36	25	13	6.3

2.4.2 Development of test conditions

To minimise the potential for binder run-off and slumping of the blocks, it was desirable to carry out the oxidation at the lowest possible temperature but it was also important to use conditions that would give results within a reasonable time. Preliminary experiments assessed the sensitivity of mix specimens to various conditions (see Table 2.3). Approximate equivalent field ages were calculated using the fitted curve in Figure 2.1. As previous research had shown that oxidation at 60°C was likely to be too slow, experiments were carried out at 85°C with both static and forced air-flow. In the latter, each compacted specimen was placed in a rubber triaxial test sleeve (100 mm diameter), one end of which was secured around tubing delivering air at approximately 2 to 3 L/min. The air was heated to test temperature before reaching the specimen by passing through several metres of copper tubing fixed to the base of the oven. The sleeve was held tightly to the sides of the specimen with rubber bands and placed on flat steel mesh to allow unimpeded air flow. These conditions allowed fresh air to be forced continuously through the specimen.

Table 2.3 Conditions used in initial oxidation experiments.

Conditions		**Time (hours)**	**Temperature (°C)**	**Conditioning**	**Viscosity at 60°C (Pas, $0.1s^{-1}$)**	**Approximate equivalent period of field ageing (years)**
A	Control[1]	-	-	-	395	-
B	Static air	90	85	None	1199	1.8
C	Static air	120	85	None	3097	3.9
D	Forced air flow	90	85	None	1427	2.2
E	Forced air flow	90	85	2 hours at 125°C in tray	2449	3.4
F	Forced air flow	90	85	5.75 hours at 125°C in tray	4295	4.6

(1) extracted unoxidised 80/100 bitumen.

Heating in static air (Table 2.3 B & C) resulted in relatively slow oxidation. Using the line fitted in Figure 2.1, up to 120 hours at 85°C produced oxidation equivalent of up to almost 4.0 years in the field. Oxidation with forced airflow (Table 2.3 D) was only slightly faster than the equivalent static air experiment (Table 2.3 B), probably because the open nature of the mix allowed rapid exchange of air even in ‘static’ conditions.

To simulate oxidation during manufacture and handling of the mix, a conditioning period was introduced. The loose mix was spread 20-30mm deep in a tray and heated at 125°C for two hours before compaction (Table 2.3 E). APRG 18 (APRG 1999) specifies conditioning at 150°C for one hour. This temperature was considered too high to represent typical plant mixing and laying temperatures for OGPA in New Zealand.

Conditioning with forced air flow resulted in oxidation equivalent to 3.4 years in the field. An additional 3.75 hours' conditioning produced ageing of about another year (Table 2.3 F).

Ageing to an equivalent field age of 4-5 years (ie. about half the average field life) was considered the minimum for reasonable durability comparisons of different mixes. The long conditioning period of treatment F is somewhat unrealistic compared with real plant mixing and handling times; and if treatment F is excluded, treatments D and E come closest to achieving the desired level. If the conditioning period is to be kept at 2 hours, a test period of 100-110 hours at 85°C with forced air flow would probably be required. However, even after 90 hours at 85° C some binder run-off occurred; so lower temperatures and a shorter (rather than longer) test period was considered desirable.

The use of pure oxygen at atmospheric pressure to accelerate oxidation rate was considered but not investigated, because it would be potentially hazardous for routine use. The oxidation rate can also be accelerated by using higher air pressures (as discussed in section 2.2); and this approach was adopted. The test temperature was reduced to 80°C, the time shortened to 72 hours to reduce binder drainage, and the air pressure increased to 2069 kPa (300 psi), increasing the oxygen concentration by a factor of 21. The loose mix conditioning time was maintained at two hours. In practice, mixing and compaction temperatures vary according to the binder in use, and no single temperature covers all situations. The appropriate conditioning temperature will depend on the binder used.

2.4.3 Final test conditions

As discussed above the test conditions adopted were:

- Conditioning of loose mix as a 20-30mm layer in a tray at 125°C for 2 hours (but in practice conditioning would be carried out using the appropriate plant mixing temperature)
- Heating of compacted specimens at 80°C and 2069 kPa air for 72 hours.

Binder drainage under these conditions was negligible.

The pressure vessel (Figure 2.2) was cylindrical (110 mm i.d. x 200 mm high) with a flat lid holding two specimens (see Figure 2.2). The lid was secured by four bolts to a flange around the upper lip of the cylinder and an air tight seal maintained with a 6 mm diameter rubber O-ring. The lid was fitted with a gas inlet valve, pressure gauge, thermocouple and a spring type safety pressure relief valve. The specimens were loaded into the vessel on a simple rack constructed from perforated galvanised plate and wire. To reduce the risk of slumping, a strip of heavy brown paper was taped around the sides of the blocks. The vessel was held at the test temperature for several hours before the specimens were introduced. The pressure inside the vessel was raised (or lowered) slowly over a period of 10 minutes using a compressed air cylinder fitted with a single stage regulator and needle valve.

The pressure vessel illustrated was originally designed for work at much higher pressures, and a simpler vessel made from mild steel pipe would suffice. The total cost of a pressure vessel, valves and fittings is likely to be in the range $1000-2000 at 2005 New Zealand prices.

Figure 2.2 Pressure vessel used to oxidise OGPA specimens.

Table 2.4 and Figure 2.3 show the effect of the test conditions on binder viscosity, with data for longer oxidation times for comparison. Oxidation for 72 hours' gave an equivalent field ageing time of 4.5 years; while 144 hours' and 216 hours' oxidation gave 6.2 and 7.5 years respectively.

Table 2.4 Effect of high pressure mix oxidation on bitumen viscosity.

Oxidation time at 80°C(hours)	**Viscosity at 60°C (Pas, 0.1s^{-1})**	**Approximate equivalent period of field ageing (years)**
72 (3 days)	4086± 1286[1]	4.5 (3.7-5.1)
144 (6 days)	9022	6.2
216 (9 days)	16410	7.5

(1) Average of 4 replicates ± 95% confidence interval.

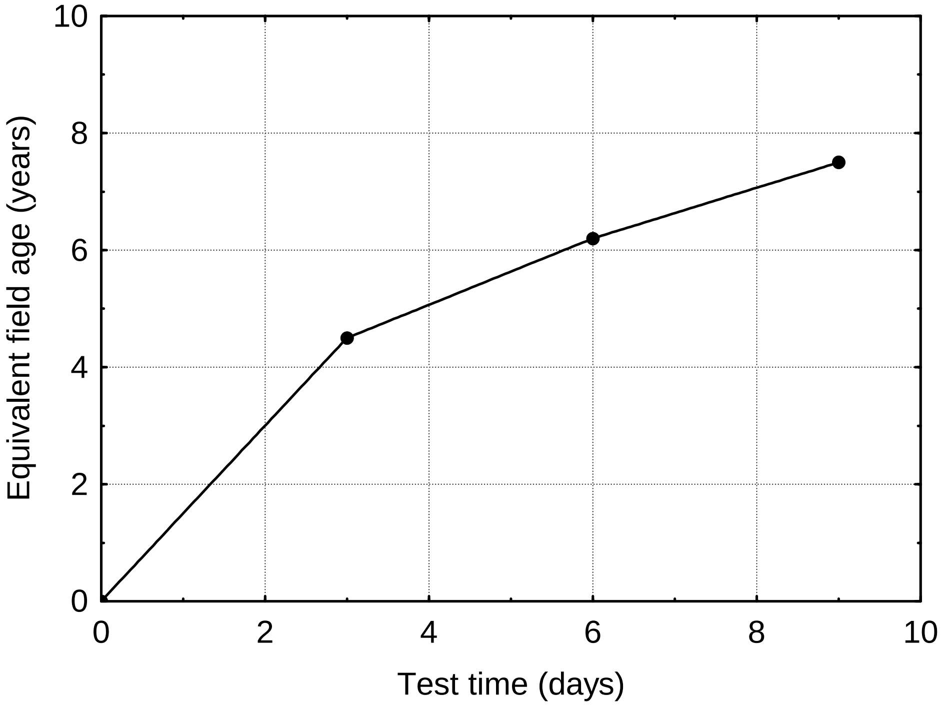

Figure 2.3 Relationship between field age and oxidation at 85°C and 2070 kPa.

High pressure can disrupt the structure of a dense mix as the pressure is raised or lowered (for example Bell et al. 1994a); but with high void OGPA this did not appear to happen. Physical disruption of the mix specimens would be apparent as a change in block dimensions after the test (see Table 2.5).

Table 2.5 Effect of high pressure mix oxidation on specimen dimensions.

Replicate	Diameter[1] (mm)			Height[1] (mm)		
	Before oxidation	**After oxidation**	**Difference (% original)**	**Before oxidation**	**After oxidation**	**Difference (% original)**
1	102.19	102.19	0	63.00	64.01	+1.6
2	102.39	102.64	+0.2	64.20	65.56	+2.1
3	102.65	102.76	+0.1	63.58	64.33	+1.1
4	102.13	102.41	+0.3	64.83	65.75	+1.4
5	102.03	102.92	+0.9	65.32	65.94	+0.9
6	102.60	102.92	+0.3	65.93	66.65	+1.1
7	101.91	102.32	+0.4	67.20	67.66	+0.7
8	102.09	101.73	-0.4	67.43	65.93	-2.2
	Average		+0.2	Average		+0.8

(1) Average of 3 measurements.

Comparison of block dimensions before and after the tests show only small average increases in height and diameter, corresponding to an average increase in specimen volume of 2% and a negligible increase in average percentage voids from 19.1 to 19.5.

2.4.3.1 Effect of oxidation on cantabro test results

Cantabro tests were carried out, using the method of Appendix A.2, on specimens oxidised as described above. Results are given in Table 2.6 and plotted in Figure 2.4. Weight losses after the test were approximately double those of the un-oxidised specimens; but increasing the test period to up to 9 days caused only relatively small further increases.

Table 2.6 Cantabro test losses of oxidised samples.

Oxidation time (hours)	**% Weight lost** (± 95% confidence limits)	**% Weight lost ratio oxidised /un-oxidised**
0[1]	12.1 ± 2.6 (n=13)[2]	1.0
72 (3 days)	25.9 ± 3.9 (n= 6)	2.1
144 (6 days)	28.3 ± 5.3[3] (n=2)	2.3
216 (9 days)	33.2 ± 4.6 (n=4)	2.7
72 Under nitrogen [4]	8.8 ± 4.0 (n=5)	0.7

(1) Conditioned for 2 hours at 125°C. (2) Number of replicates. (3) Range. (4) At 2070 kPa (see text).

To further explore the effect of pressurisation without the complicating effect of oxidation, some specimens were treated in an inert nitrogen atmosphere. The test was done as normal, except that to purge the vessel (and specimens) of oxygen it was pressurised slowly to 50 psi with oxygen-free nitrogen and then depressurised. This process was repeated three further times before filling the vessel to 2070 kPa (300 psi) with nitrogen over 10 minutes as usual. Assuming air to contain 21% oxygen and atmospheric pressure to be 101 kPa (14.7 psi), this procedure leaves less than 0.01 % by volume of oxygen in the vessel

Cantabro test results (Table 2.6) for the nitrogen treated specimens are not significantly different (95% confidence level) from those of untreated specimens. This indicates that higher Cantabro test losses in oxidised specimens are due to oxidation of the bitumen rather than being artefacts of the procedure arising from bitumen run-off or pressurisation damage to the mix structure.

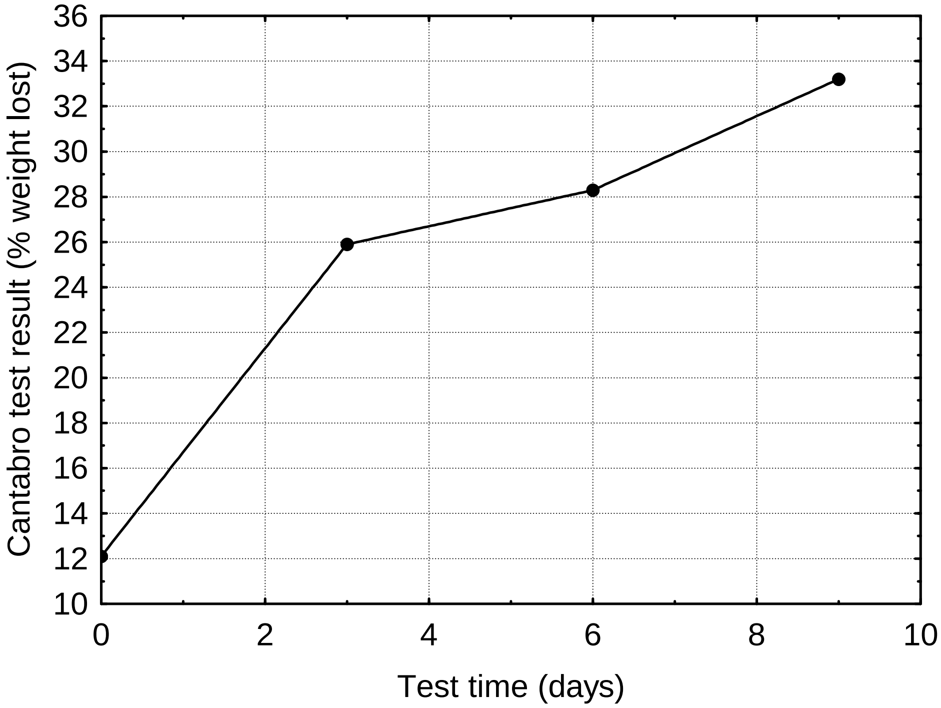

Figure 2.4 Effect of oxidation at 85°C and 2070 kPa on cantabro test losses at 25°C.

3. Application of the test to plant mix

To compare the results of the test procedure on laboratory mixes with data from actual plant mixes, samples of OGPA were taken from an asphalt plant during normal plant operation over a period of several weeks. Specimens of two mix types were prepared by compaction at the plant mixing temperature and put through the test as described above, but without the conditioning treatment. Mix A was a standard PA 14 type and mix B was a PA 14HS type, both using 80/100 bitumen without modifiers. Mix properties are given in Table 3.1.

Table 3.1 Plant mix grading curves (% passing)

	Sieve size								
	13.20	**9.50**	**6.70**	**4.75**	**2.36**	**0.60**	**0.075**	**Binder[1] content (%)**	**Air[1] voids (%)**
Mix A (PA 14)	100	95	57	21	14	7.4	3.4	5.1±0.1	19.2±0.8
Mix B (PA 4HS)	100	91	68	33	23	12	4.5	5.1±0.1	15.4±0.8

(1) ±95 % confidence limits

Cantabro test results for both mixes before and after oxidation (72 hours) are given in Table 3.2. Both mixes gave cantabro test losses (both before and after oxidation) less than that of laboratory prepared mix (see Table 2.6), although the ratios of the losses are similar to those found with the laboratory mix.

Table 3.2 Cantabro test losses for oxidised plant mix.

Mix	**% Weight lost**		**Ratio oxidised /un-oxidised**
	Un-oxidised (± 95% confidence limits)	**Oxidised (± 95% confidence limits)**	
A	7.3 ± 0.7 (n= 54)[1]	13.7 ± 5.3 (n=6)	1.9
B	9.0 ± 1.8 (n=9)	13.3 ± 2.5 (n=10)	1.5

(1) Number of replicates.

4. Conclusion

A simple, inexpensive laboratory test has been developed to assess the durability of OGPA in the field. It is intended mainly for use in mix design rather than for quality control during production runs. The test involves conditioning loose mix at 125°C (or the appropriate plant mixing temperature) for two hours to simulate oxidation during manufacture and handling. The conditioned mix is compacted into standard Marshall test sized specimens, which are heated at 85°C for three days under 2070kPa air pressure and then subjected to the cantabro test at 25°C.

Viscosity measurements on 80/100 bitumen from field samples suggest that the test simulates approximately 4.5 years' field ageing, about half the average lifetime of OGPA in New Zealand. This is enough for meaningful conclusions to be reached about the relative durability of different mixes. In contrast to results reported in the literature for denser graded materials, the use of high air pressure appears to cause no significant disruption to the OGPA mix structure. Without using high pressure air, the test would take an impractically long time unless much higher temperatures were used, probably resulting in binder runoff.

For the three 80/100 bitumen mixes studied, the percentage weight loss of the oxidised mix specimens in the cantabro test increased by 1.5 to 2 times compared with un-oxidised specimens. After oxidation the absolute loss was highest for laboratory prepared mix at 26%, while the two plant mixes gave losses of 13-14%. On this basis a limit for acceptable loss after the test could be set at 30%. Based on the three materials studied, an appropriate design criterion for cantabro losses of unoxidised mixes would be 15%. These figures are tentative and should be confirmed by testing of additional mixes. By comparison APRG 18 (APRG 1999) suggests cantabro loss values of 20-25% for un-aged mix. It provides no long term ageing procedure.

The cantabro test was chosen to measure the effect of mix oxidation partly for its simplicity, and also because it is already widely used in New Zealand and internationally to measure the cohesion and abrasion resistance of OGPA. Except for the laboratory prepared mix, the increases in weight loss after oxidation were small. Losses after extended periods (6 and 9 days) of oxidation were not much greater, despite large increases in binder viscosity. Cantabro losses increased by a factor of 1.3 when the oxidation time increased from 3 to 9 days compared with a factor of four for the viscosity. These observations suggest that the severity of the test may need to be increased, most simply by increasing the number of cycles. Cantabro test results for the two plant mixes were very similar, suggesting either that (if the PA and PA14HS TNZ specifications do actually result in different tensile strength mixes) mix cohesion is not closely related to indirect tensile strength, or that the cantabro test is a relatively insensitive measure of cohesion.

The Cantabro test is normally performed using three replicates, but the repeatability of the test at 25°C does not seem to have been published in a standard specification. The precision of the test improves with a greater number of replicates but this must be offset against the increased cost involved.

5. Recommendations

The durability test should be trialled with a larger number of different mixes in at least two laboratories to:

- establish acceptable limits for cantabro percentage weight loss;
- determine whether separate limits are required for 60/70 and 80/100 bitumens;
- determine whether the cantabro test is sensitive enough to satisfactorily distinguish differences in durability, or whether an alternative measurement of cohesion (e.g. Marshall flow) is desirable;
- establish the repeatability and precision of the test.

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7. Appendices

Appendix A1 Method for the extraction of bitumen from asphalt mix.

The sample was warmed at 60°C for 0.5-1 hour until soft enough to break easily into small lumps. When cool, 75 ml of dichloromethane (A.R. grade) was added to 100g of sample and left with occasional stirring for one hour, covered and in the dark.

The solution was passed through a 150 micron sieve and the aggregate washed with 2x 10 ml fresh dichloromethane. After centrifugation for 20 minutes at 2000 rpm (939 g) the bitumen was filtered under vacuum (water pump) through Whatman grade 1 and GFC filters (grade 1 paper on the bottom).

Approximately equal portions of the solution were poured onto polished 245 x 340 mm, stainless steel plates. Stainless steel was used instead of glass to reduce the possibility of selective surface adsorption of polar species. A wide bladed spatula was used to spread the solution, allowing the solvent to evaporate and leave a thin film of bitumen.

The plates were immediately placed in an airtight cabinet (approximately 60 L) in the dark and purged with nitrogen at >10 L min^{-1} for ten minutes, after which time the purge rate was decreased to about 1-2 L min^{-1}. After 50 minutes the plates were removed and the bitumen scraped off with a single sided razor blade.

The last traces of solvent were removed by heating the combined bitumen scrapings (about 3g) at 100°C for 30 minutes under >29.9” Hg vacuum. The samples were stored at 4°C.

Viscosity measurements were made using a cone and plate geometry on a Carrimed Instruments CSL 500 rheometer fitted with a water bath for temperature control of the specimen.

To determine whether the procedure significantly affected the viscosity of the recovered bitumens, samples of 80/100 bitumen were ‘extracted’ using the procedure and the initial and post-extraction viscosities compared (see table A1.1).

Table A1.1 Effect of extraction procedure on bitumen viscosity (60°C, 0.1 s^{-1}).

Viscosity (Pas)	
80/100 bitumen	**80/100 bitumen after extraction**
444	427
376	400
410	320
Mean = 397	395

Appendix A2 Cantabro Test Procedure and Mix Compaction

The Cantabro test procedure and detailed specifications for equipment are given in APRG Report 18 (APRG 1999), which is in turn based on AS 1141.23 (AS1141.23). A similar procedure is given in AST 07 (Austroads 2003). Right cylinders of compacted mix (100 mm diameter and 50-70 mm high) were brought to a temperature of 25± 0.5 °C in an incubator and then tumbled in a steel drum (maintained at 25±3 °C) for 300 revolutions at 30 rpm. The weight of aggregate lost from the specimen through abrasion was recorded as a percentage of the original weight.

Specimens were allowed to stand at room temperature for at least 24 hours before testing. Specimen temperatures were monitored using a dummy block of mix drilled to take a thermocouple. At least five hours (usually overnight) were allowed for specimens to stabilise at the test temperature. The test machine was enclosed in a large cabinet through which temperature controlled air from a refrigeration-heating unit was circulated.

The repeatability of the test as set out in APRG 18 does not appear to have been established.

Asphalt mix compaction

All mixes were compacted with 75 blows of a mechanical Marshall hammer. Conditioned laboratory prepared mixes were compacted at the conditioning temperature, 125°C. Plant mixes were compacted at the plant mixing temperature.