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Table of Contents

1. Introduction

1.1 Purpose of Report

In November 2013, the Arup URS Consortium was commissioned by the Highways Agency to carry out a programme of work on "Optical Spectroscopy of Binder Condition at Traffic Speed - Feasibility Study", under the Department for Transport (DfT) Framework for Transport Related Technical and Engineering Advice and Research – Lot 2: 4/45/12, Package Order reference: 223(4/45/12)ARPS.

Following the inception meeting on 10th December 2013 at the URS Nottingham office, which was attended by Pejman Dehdezi, Daru Widyatmoko, Jessica Tuck (URS), Martin Heslop (Acland) and Donald Burton (Highways Agency), it was confirmed that the work comprised four sub-tasks:

- Sub-Task 1: Literature review
- Sub-Task 2: Likelihood of success of a research programme
- Sub-Task 3: Identify a research programme
- Sub- Task 4: Develop a proposed research programme

The principal aim of this work is to carry out a feasibility study to identify the extent and duration of a programme of research to develop a traffic speed tool to monitor age-related changes in asphalt mixtures surfaces.

2. Background

The total road length of highway networks in the UK was estimated to be 245,000 miles in 2010 [1], and had increased by approximately 1% per year since 2000. This demonstrates that the highways authorities have shifted the attention from new construction to the maintenance and rehabilitation of the existing network.

According to the Annual Local Authority Road Maintenance Survey [2], in England, the government has spent heavily on road maintenance in recent years. £2,240m was spent in 2006, £937m in 2007 and another £861m in 2008, an aggregate total of £6,867m from 2002 to 2008, in order to maintain the serviceability level of pavements. The survey also reports that a further £10,650m is currently required to bring the UK's roads to the desired standard. In addition to the expenditure on carrying out the work, the travel delay cost to the road users caused by maintenance is significant and expected to substantially exceed the corresponding cost of maintenance.

As highways agencies have turned their attention from construction of highway networks to pavement maintenance and rehabilitation (M&R), the need for optimal M&R strategy is emerging. A typical life cycle of a pavement is presented in **Figure 1** where pavement condition cycles are shown for a preventative maintenance trigger and for a rehabilitation trigger [3]. Maintenance treatments can never fully restore the pavement condition to the original performance and the effectiveness of a surface treatment in delaying rehabilitation depends when it is carried out, i.e. at what stage of deterioration. This is currently assessed by visual inspection and in many cases is thought to be too late, with structural damage having commenced. Other maintenance triggers include skid resistance and defects such as fretting and ravelling (sometimes picked up by increasing macrotexture). In order to establish the preventative and rehabilitation triggers to maximise their effectiveness, technologies are needed that can detect pavement distresses early in a deterioration cycle, preferably before significant damage is visible. Methods of examining the pavement condition have historically not included monitoring of the binder condition owing to the difficulty of obtaining samples for test. Coring of road pavements is both laborious and carries safety risk; extracting the binder using solvents to see how it has aged in a small area of the network does not provide an adequate monitoring tool. A previous project to examine the effectiveness of preservatives provided a successful method of extracting aged binder from the surface of roads and airfields using high pressure water jetting as shown in **Figure 2** [4]. However, although the protocol provided samples from large areas quickly compared with coring, the current system is not able to be carried out at traffic speed. Donald Burton (Highways Agency) considered that a feasibility study to evaluate Optical Spectroscopy was a way forward.

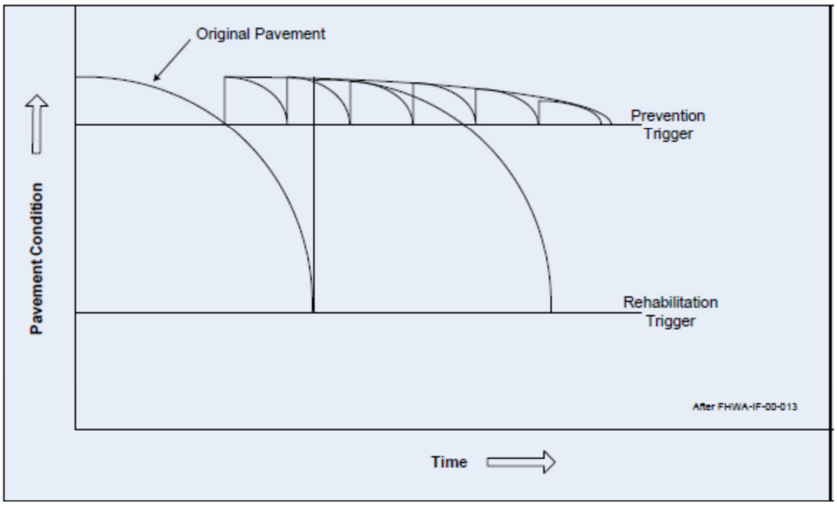

Figure 1 Pavement preservative scheme based on preventative maintenance [3]

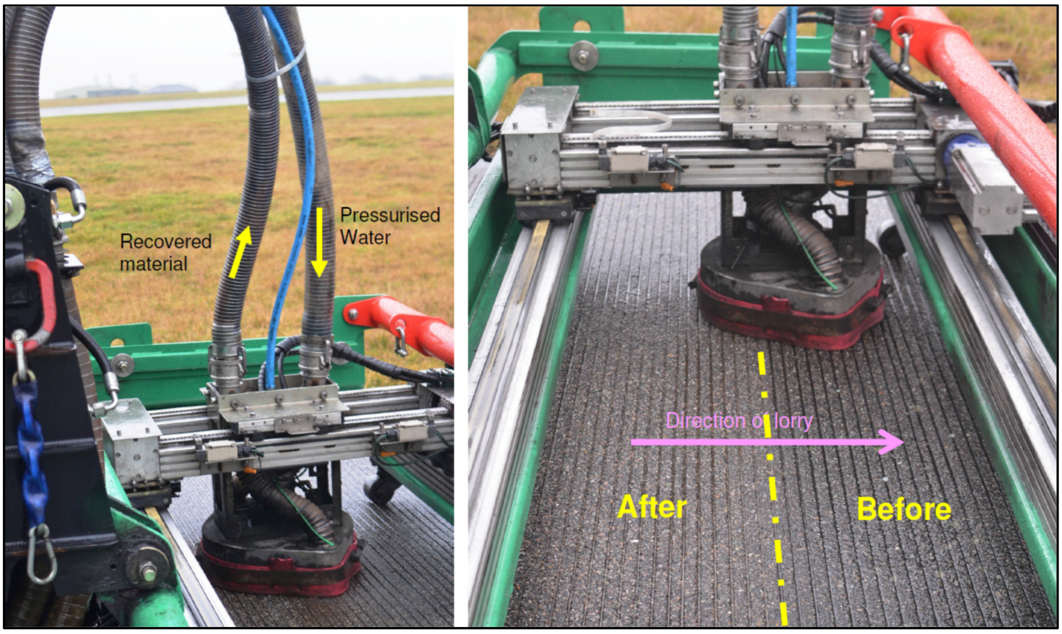

Figure 2 Sample recovery Process [4]

3. Scope

Considering the time and financial constraints, it was agreed with the HA Client Package Order Manager that the assessment would be carried out by a desk study, as shown in **Figure 3**.

This report reviews published literature on the use of spectroscopy techniques to detect the age hardening of the binder.

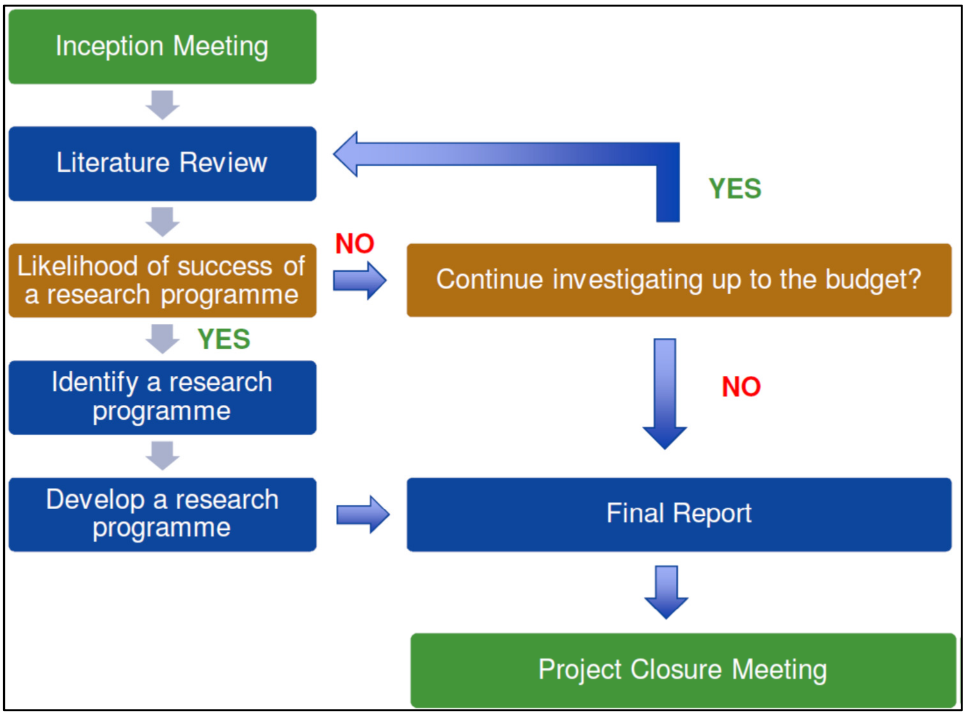

Figure 3 Approach to the study

4. Ageing of the Bitumen

Age hardening of bitumen is one of the main factors that significantly affects the durability of bituminous paving materials. The asphalt mixture will become brittle as a result of age hardening, and its ability to support traffic loads will significantly reduce. In addition, excessive hardening can also weaken the bond between the bitumen and aggregate, resulting in loss of materials at the surface layer.

4.1 Main Factors Affecting Ageing

Three fundamental composition-related factors which govern the changes that could cause bitumen hardening in pavements are as follows [5]:

- Loss of oily/volatile components from the bitumen to the air and/or aggregates,
- Change in chemical composition of bitumen molecules from reaction with atmospheric oxygen,
- Exudative hardening due to filler, aggregate porosity and bitumen properties and,
- Molecular structuring that produces thixotropic effects (steric hardening).

4.2 Molecular Changes in Oxidation

The chemical functional groups formed through oxidative ageing include sulphoxides, anhydrides, carboxylic acids and ketones (anhydrides, carboxylic acids and ketones are normally classified into carbonyl functional group) [5]. Ketones and sulphoxides are the major oxidation products formed during oxidative ageing; hence, special attention is paid to the formation of ketones and sulphoxides [5]. **Figure 4** illustrates the structural formulae of important chemical functionalities in bitumen that are normally present or formed on oxidative ageing [5].

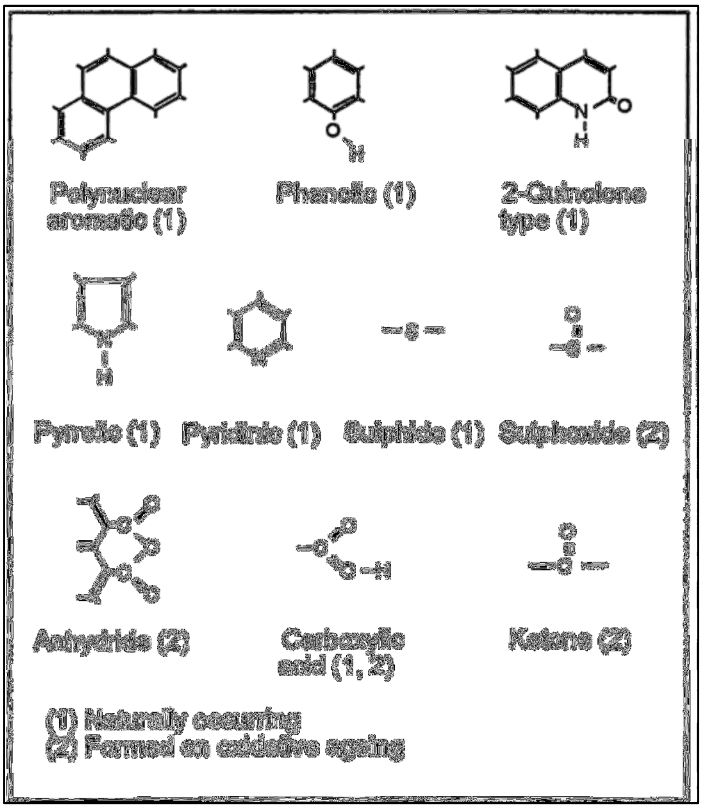

Figure 4 Chemical functionalities in bitumen molecules normally present or formed on oxidative ageing [5]

4.3 Laboratory Tests for Ageing Study

There are several existing experimental techniques that could be used to study the effect of ageing on the performance of bituminous paving materials. However, these techniques are destructive and require asphalt samples to be collected from the site; hence, they require road closures (traffic disruption) and testing in a laboratory. Some of the techniques are tabulated in **Table 1**. Among them the Fourier Transform Infrared Spectroscopy (FTIR) technique has the potential to be considered as an option for field testing. This is due to its potential application as a contactless and dynamic tool, which is necessary to satisfy the research objective (i.e. Optical Spectroscopy of Binder Condition at Traffic Speed).

Table 1 Some of the techniques to study age-hardening of bitumen [5]

Test	Method	Outcome	Application (Field/Lab)
Dynamic Shear Rheometer (DSR)	Apply an oscillatory shear force to a bitumen sample	Complex shear modulus (G^*), Phase Angle (δ)	Laboratory
High Pressure Gel Permeation Chromatography (HP-GPC)	Measure the molecular size distribution of the un-aged and aged bitumen	Molecular size distribution	Laboratory
SARA Analysis	Analyse the chemical composition of bitumen	Quantity of Saturates, Aromatics, Resins, and Asphaltenes in bitumen	Laboratory
Differential Scanning Calorimetry (DSC)	Measure the enthalpy of bitumen at different temperatures	Enthalpy-Temperature curve of bitumen	Laboratory
Fourier Transform Infrared Spectroscopy (FTIR)	Energy from the infrared light source is transformed into vibrational energy in the molecules of bitumen.	Characterise the oxygen-containing functionalities in the bitumen	Laboratory/Field

5. Principle of Spectroscopy

The principle of spectroscopy is briefly summarised in this section, prior to review of the different technologies that are commonly used to fingerprint construction materials.

Spectroscopy is a powerful technique for characterising physical materials using the principle of the varying absorption (or emission) of different energy wavelengths. The samples may be material in phase: solid, liquid, gas, or plasma, and may be light emitting or light absorbing [6].

The energy used in spectroscopy may lie within the range of wavelengths visible light, or may be in the infrared or ultraviolet regions of the electromagnetic spectrum. Spectroscopy requires the diffraction of energy into a rainbow of wavelengths, so that the variation in light intensity versus wavelength can be measured (and usually also recorded).

Spectroscopy employs a dispersive optical element to spread the spectrum of light into spatially separated wavelengths. Prisms are sometimes used, but diffraction gratings are more commonly employed, because of their higher dispersion, and their ability to be optimized for a wide range of optical wavelengths. There are several optical and physical arrangements used in spectroscopy.

With regard to age hardening, FTIR is widely used to characterise the oxygen-containing functionalities in the bitumen. Molecular bonds in the bitumen can vibrate at different frequencies depending on the molecule, chemical environment and the type of the bonds. In the FTIR test, the energy from the IR light source, with wavenumbers of 400 to 4000 cm^{-1}, is transformed into vibrational energy in the molecules of bitumen. It is this vibrational energy that presents a series of absorption bonds which are recorded as transmittance (%) or absorbency against the wavelength (μm) or more often wavenumber (cm^{-1}). The positions of the peaks along the wavenumber axis are unique to certain chemical bonds and chemical functional groups present in the bitumen. Typical wave numbers for several bitumen ageing products are as follows [5]:

- S=O, C-H in aromatic - 1020 cm^{-1}
- SO2 from oxidation - 1108 cm^{-1}
- ester (R-COOR) - 1262 cm^{-1}
- C=C in aromatic - 1618 cm^{-1}
- C=O in carbonyl - 1702 cm^{-1}

5.1 Some Common Spectroscopy Techniques:

Attenuated Total Reflectance FTIR (ATR-FTIR): This is a direct sample contact technique for measuring the spectra of solids, semisolids, liquids, and thin films (see **Figure 5**). The key component of the ATR techniques is an IR transparent crystalline material with a high refractive index (e.g., diamond, zinc selenide, germanium). The crystal shape is cut to mimic a waveguide cavity; however, the ends of the rectangular solid are bevelled. The IR source enters one end of the crystal, and the bevel surface refracts the beam toward the top of the crystal surface where it is reflected toward the bottom of the crystal. At this point the IR energy extends several microns beyond the bottom surface and interacts with the sample below. The reflected wave is then reflected to the top of the crystal, then toward the other bevel end of the crystal, and then to the detector. Typically ATR accessories interact with the top 0.1 – 0.5 micron of surface depth penetration to collect spectral data.

Figure 5 Attenuated Total Reflectance FTIR (ATR-FTIR)

Diffuse Reflectance handheld spectroscopy (DR-FTIR): this is a technique that uses the “modulated” IR energy from the spectrometer to illuminate a solid sample and collect the diffused reflected IR energy from the sample and focus this energy onto the detector. It is a non-direct contact and the device can test at a distance of 0.5 to 1 cm from the sample (see **Figure 6**).

Figure 6 Diffuse Reflectance handheld spectroscopy

Photoacoustic spectroscopy (PA-FTIR): This FTIR accessory technique detects IR absorption by an acoustical transducer process. As a sample absorbs IR energy in a contained vessel, it heats up on a molecular level. As the sample is irradiated at a specific FTIR optical path difference (OPD) instance, the heat is transferred from higher temperatures to lower temperatures, meaning that the heat creates a thermal wave that moves toward the surface. Once the wave arrives at the surface, the gas above the sample is heated and then expands. Usually helium is used for the gas although air may be substituted. The gas expansion causes a pressure wave to propagate through the gas volume. At the top of the closed sample vessel is a highly sensitive microphone that produces a signal from the time-varying pressure waves that are directly related to the absorption at a given FTIR OPD instance. In fact, a plot of the microphone-signal variation output vs the OPD position results in an interferogram. Various approaches for depth-profiling may be used. An absorption spectrum may be computed via a Fourier Transform similar to other FTIR processing approaches. The PA-FTIR does require a sample preparation process to be placed in the acoustic containment vessel, and therefore implicitly requires contact with the sample material.

Transmission FTIR (T-FTIR): The test sample is placed in the optical path between the output of the FTIR and the detector. In this configuration, the “modulated” IR beam passes through the sample and the spectral absorbance (or conversely transmission) is measured. Since the IR energy cannot pass through solids, the solids must be reduced to small particles that are suspended in an inert material such as potassium bromide (KBr). Using this approach, the IR energy may interact with the particle’s molecules, while the inert suspension material allows the IR energy to propagate through the sample volume to the detector side. In this case, considerable sample preparation is required for the sample vessel. A liquid or gas sample may be filled in a sample vessel and the IR light propagates through the vessel to the detector.

5.2 FTIR Analysis

Figure 7 shows an example of the FTIR scan results for an un-aged binder and the same binder aged by different ageing simulation methods [5]. The regions for the main oxidative ageing products are: Carbonyls (1700 cm^{-1}) and sulphoxides (1030 cm^{-1}). From the Figure, it can be seen that the absorbance values at wavenumbers representing carbonyl and sulphoxide both increase as the ageing simulation proceeds, which indicates that the amount of oxidation products in bitumen increased during the ageing simulation.

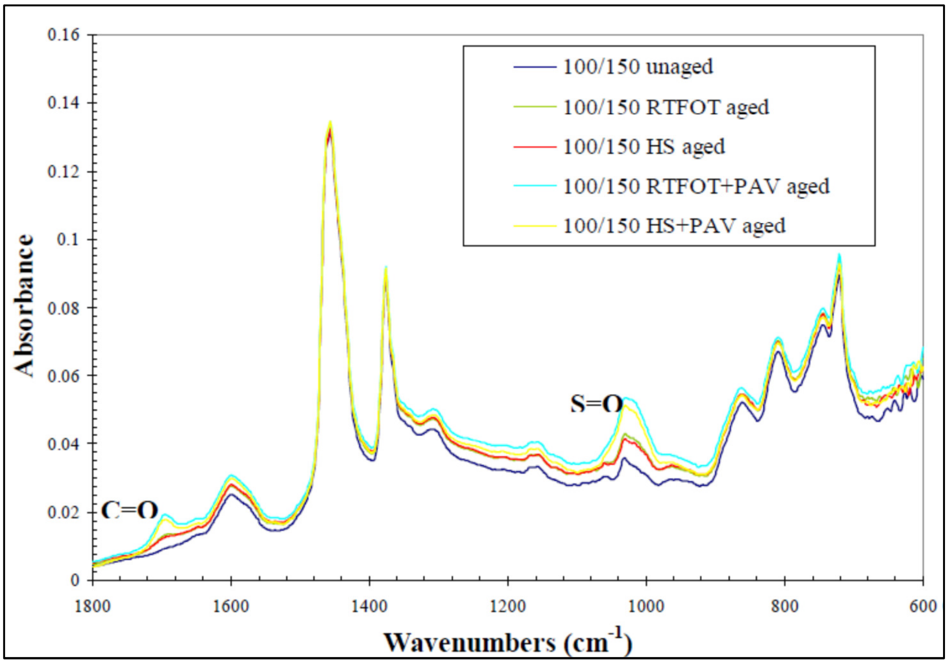

Figure 7 FTIR results for binders [5]

6. Literature Review of Field Spectroscopy

All the tests mentioned in Section 4.3, with the potential exception of FTIR, require physical samples to be taken and hence disruption to the traffic in order to collect site samples for subsequent laboratory testing. Various studies employed FTIR to investigate aging of asphalt. For instance, FTIR was used to quantify the extent of oxidation based on the area under the carbonyl absorption band [7-12]. FTIR has also been used to detect the presence of a stripping agent [13, 14]. AASHTO T302-05 standardized the FTIR method to identify polymer-modified binders (PMB) procedures [15].

The Previous attempts to perform field spectroscopy on the condition of binder have been extremely limited, but there have been two studies in the US.

6.1 Western Research Institute (WRI)

Western Research Institute (WRI) [16] has carried out a study, published in 2010, which aimed to:

- Predict age-related embrittlement in asphalt pavement surfaces;
- Develop ground-based and airborne systems to measure key spectral indicators needed for prediction.

The WRI idea was to predict failure so that pre-emptive measures could be applied to the surface to extend the pavement life. The project involved five tasks as follows:

- The first task was concerned with determining the infrared spectral features of ageing asphalts that best correlate with physical properties, and determining whether spectral manipulation methods could isolate the asphalt signature from that of aggregates.
- The second task examined field cores, mapped ageing severity with depth, and examined surface ageing.
- The third task developed a ruggedized FTIR, an instrumentation vehicle, and supporting software to implement field analyses.
- The fourth and fifth tasks were to be field trials with completed systems.

Some of the findings from the WRI study are summarised below.

- WRI used two technologies to establish a relationship between carbonyl content and the complex modulus of the bitumen (G*) in the laboratory. A Transmission FTIR (T-FTIR) and a photoacoustic FTIR (PA-FTIR), which both require direct contact with bitumen, were used. **Figure 8** shows that there is a good relationship between G* and carbonyl content. In addition, a comparison of carbonyl peak height from T-FTIR and PA-FTIR revealed a reasonably linear relationship between these two techniques (see **Figure 9**).

  These results provide some validation of the concept that rheological properties of asphalt binders can be estimated from changes in asphalt chemistry measured by FTIR.

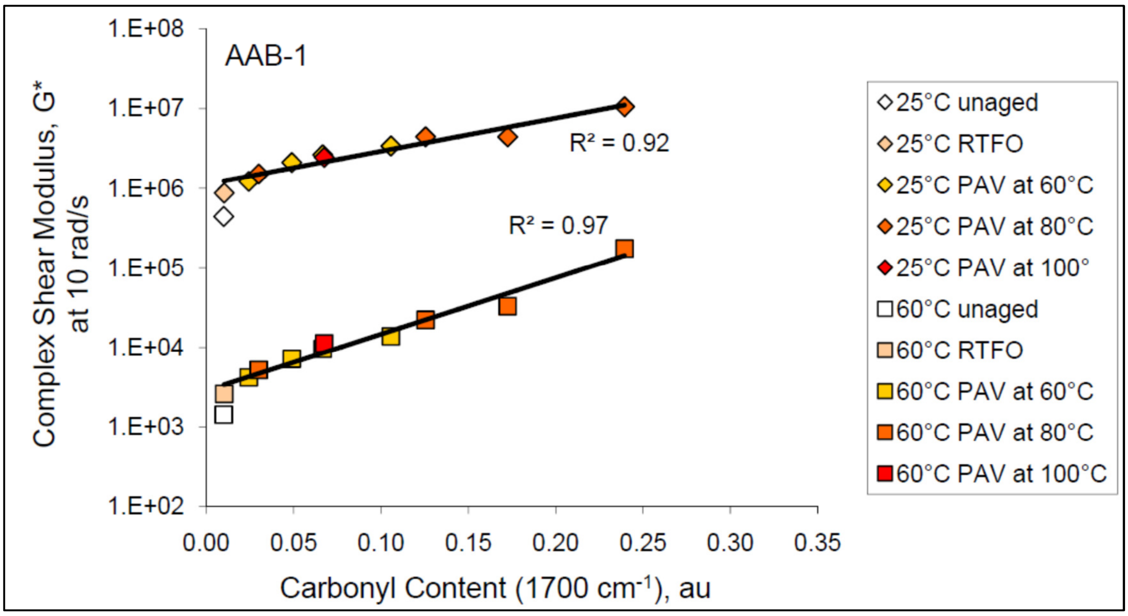

Figure 8 Correlations of complex shear moduli with carbonyl content, using a Transmission FTIR [16]

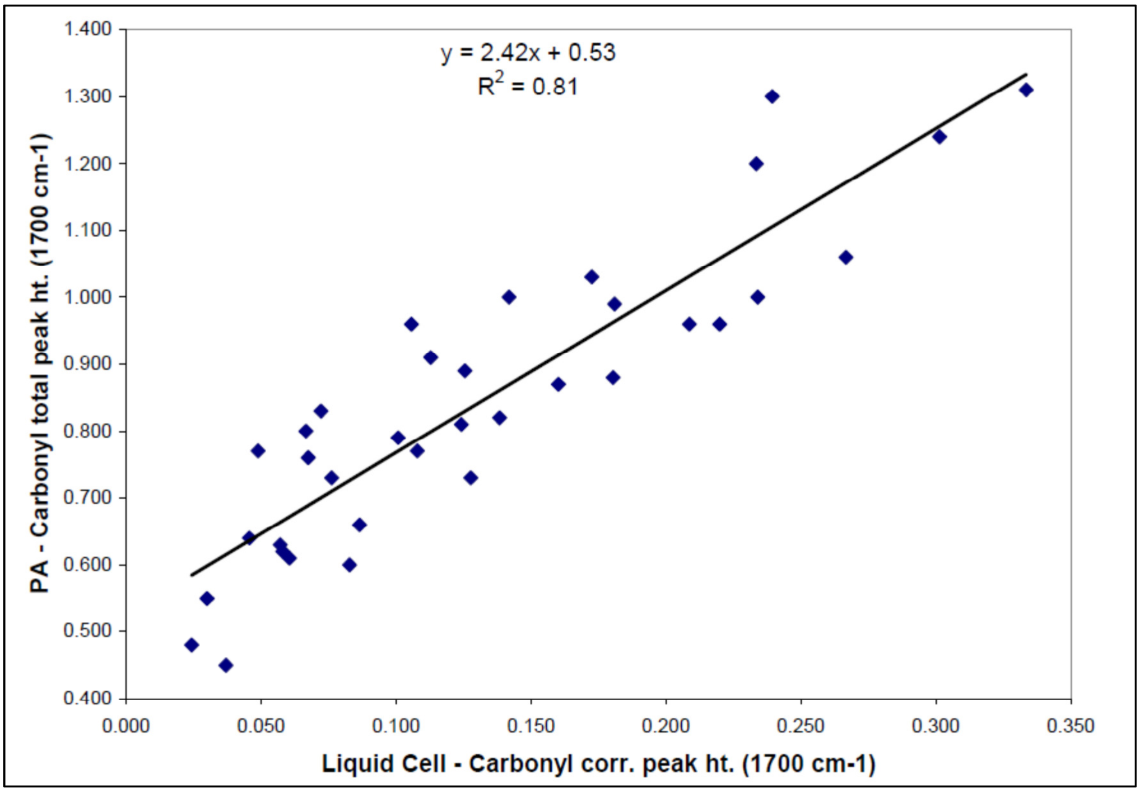

Figure 9 Carbonyl peak height at 1700 cm^{-1}, PA-FTIR vs. T-FTIR [16]

- The degree to which the aggregates can affect the spectrograph of the carbonyl absorption signature of an asphalt mixture was investigated by the WRI [16]. It was found that the PA-FTIR spectrum of pure aged bitumen can be isolated from the spectra of aged mastics of bitumen and aggregates in the laboratory (see **Figure 10**).

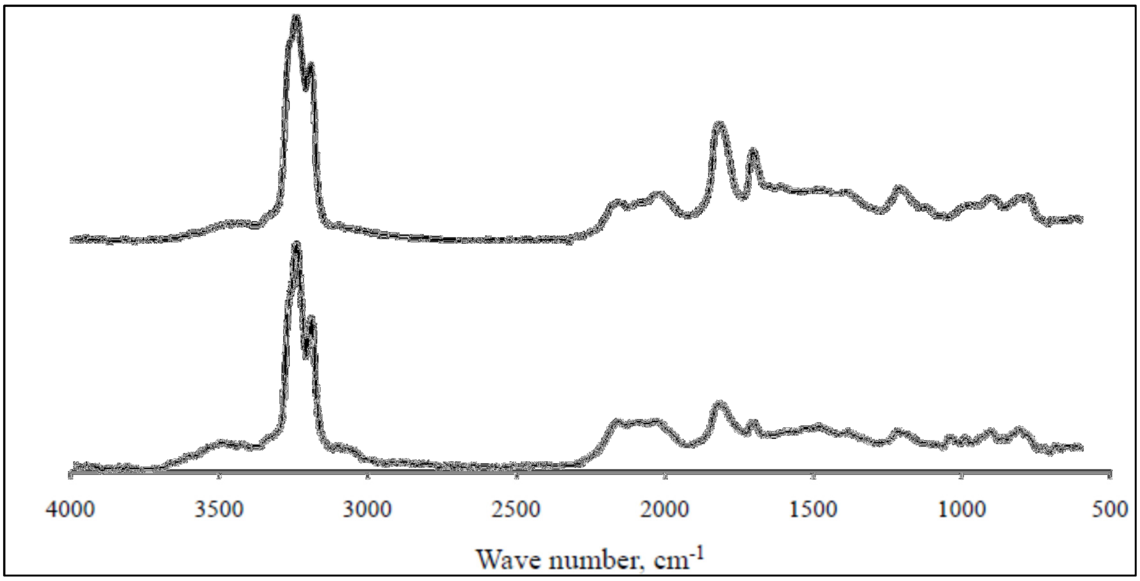

Figure 10 Comparison of pure aged bitumen spectra (top) with that of bitumen extracted from mastic (bottom) [16]

- One of the challenges of field spectroscopy for this application is that the presence of atmospheric carbon dioxide could affect the spectrum of the carbonyl absorption signature of an asphalt mixture. WRI [16], through laboratory testing, concluded that there is sufficient signal to identify atmospheric carbon dioxide. The infrared absorption region around 1700 cm^{-1} is not an "atmospheric window" for remote sensing. Moisture in the atmosphere strongly absorbs in this region, so for remote sensing applications another part of the spectrum must be used.
- For monitoring ageing severity, the region around 1200 cm^{-1} has little atmospheric interference and the absorbance in a broad band near 1200 cm^{-1} correlates well with the complex shear modulus [16].
- The PA-FTIR technique provides the best spectra, but needs significant method development to be adapted for field use.

Although the WRI study overcame many of the challenges of field spectroscopy, the field validations (i.e. Fourth and Fifth tasks) could not be completed due to the time and budget constraints.

6.2 Second Strategic Highway Research Program (SHRP2)

This SHRP report [17] studied the evaluation of portable and stationary spectroscopy devices and their capabilities to "fingerprint" typical construction materials. The portable spectroscopic techniques evaluated included Attenuated Total Reflectance (ATR) FTIR, Raman, X-Ray Fluorescence (XRF), and X-Ray Diffraction (XRD). The stationary spectroscopic techniques evaluated included Nuclear Magnetic Resonance (NMR) and Gel Permeation Chromatography (GPC). **Figure 11** shows a summary of the portable equipment used in the SHRP2 study.

Feature[a]		Target Value	FTIR	Raman	XRF	XRD
Accuracy	Minimum	1%	<0.5%	<2%	<1%	<1%
	Goal	<0.5%				
Duration of measurement	Maximum	1 h	~1 min	~1 min	6–12 min	15 min
	Goal	~5 min				
Effort involved	Maximum	1 person	1 person	1 person	1 person	1 person
	Goal	1 person				
Amount of prior training	Maximum	1 day	1 h	1 h	1 h	1 h
	Goal	0.5 day				
Reliability	Minimum	Depends on material (90%)	99% (software failure)	Depends on material[c]	99% (software failure)	99% (software failure)
	Goal	95%				
Time to get results	Maximum	Depends on construction process (1 h)	~5 min	~5 min	~5 min	~5 min
	Goal	~5 min				
Price range	Maximum	$50,000	~$25,000	~$60,000	~$37,000	$45,000
	Goal	<$20,000				
Device weight	Maximum	50 lb	~16 lb	~20 lb	~4 lb (handheld) ~15 lb (benchtop)	~27 lb
	Goal	<20 lb				
Sample preparation	Maximum	Solvent	As is[b]	As is[b]	As is (liquids) pulverization (solids)[b]	Crushing (solids)
	Goal	As is[b]				

Figure 11 Summary of the portable equipment used in SHRP2 [17]

The materials list included epoxy coatings and adhesives, traffic paints, Portland Cement Concrete (PCC) with chemical admixtures and curing compounds, asphalt binders, emulsions, and mixes with polymer additives. **Figure 12** summarises the spectroscopic methods in terms of their success for each mixture. ATR-FTIR analysis performed well for all cases except to detect diesel contamination in asphalts. The stationary GPC and NMR systems were successful in the detection of all additives in asphalt products except anti-stripping agents (most likely because of their extremely low concentration in the mixtures).

With regard to the stationary devices, NMR imaging facilitated assessing the degree of aging and asphalt compatibility in aged asphalt [18]. NMR imaging was also recently used to evaluate moisture-induced damage in asphalt layers by measuring the interfacial properties of asphalt components [19]. NMR has traditionally required a laboratory environment because of the high magnetic fields required. Recently, however, portable NMR devices have become available [20], creating an excellent opportunity to explore their application in field conditions as part of this study. GPC can be used to study the rheological properties of asphalt, such as viscosity and resistance to oxidation and also the aging of asphalt based joint sealants [21-24].

Material Category	Objective	Portable Methods				Stationary Methods	
		FTIR	Raman	XRF	XRD	GPC	NMR
Epoxy coatings, paints, and adhesives	Presence of solvents	Yes	na	na	na	No	Yes
Waterborne paints	Presence of water	Yes	na	na	na	na	na
PCC	Identification of admixture in PCC mix	Yes[a]	na	No	na	na	na
	Quantification of content	No	na	No	Yes[b]	na	na
Curing Compounds for PCC	Identification of curing membrane on PCC surface	Yes	na	na	na	Yes	na
Polymer-modified asphalt binders, emulsions, and mixtures	Identification of polymer and water in product	Yes[b]	No[c]	na	na	Yes	Yes
	Quantification of content	Yes	na	na	na	Yes	na
HMA concrete	Detection of contaminants (e.g., motor oil, diesel fuel)	No	No[c]	na	na	Yes	Yes
Antistripping agents in binders and mixtures	Identification of antistripping in product	No	No	na	na	No	Yes
	Quantification of content	No	No[c]	na	na	No	na
Oxidation in RAP	Verification of presence in mixture	Yes	No[c]	na	na	Yes	Yes
	Quantification of content	Yes[c]	na	na	na	Yes[c]	na

Note: na = not applicable.
[a] For concentrations greater than 0.4%.
[b] High variability in results is expected.
[c] Not applicable for solids and fluorescing constituents.

Figure 12 Summary of success in spectroscopic identification and quantification of additives and contaminants [17]

The SHRP2 study concluded that a handheld FTIR can be potentially used on construction sites, especially for the analysis of asphalt and concrete products. However, due to the time and budget the device was not available to the SHRP team.

6.3 Laser-Induced Remote Sensing

The Laser-Induced Breakdown Spectrometer (LIBS) instrument uses powerful laser pulses, focused on a small spot on target rock and soil samples within 7 m of the rover, to ablate atoms and ions in electronically excited states from which they decay, producing light-emitting plasma. The LIBS can rapidly identify the kind of rock being studied, determine the composition of soils, and measure the abundance of all chemical elements.

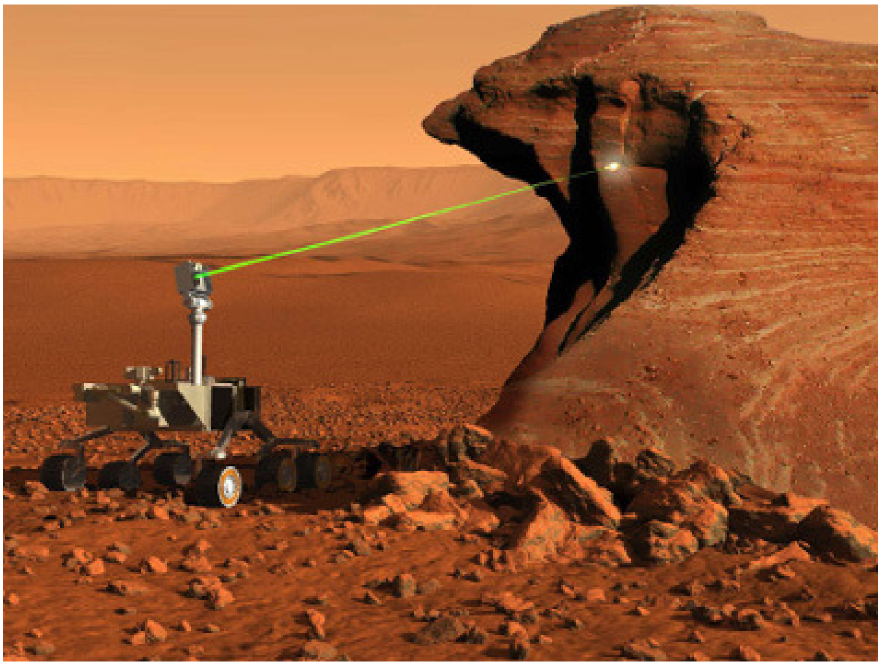

Figure 13 Laser-Induced Breakdown Spectrometer (LIBS) [25]

Understanding the technical details incorporated in the LIBS could help to achieve a non-contact device to measure and quantify the chemical element in the asphalt mixtures.

7. Challenges and Likelihood of Success of a Research Programme

From the literature review, FTIR spectroscopy has been identified as one of the most promising spectroscopic methods for “fingerprinting” construction materials including asphalt concrete. The FTIR spectroscopy technique was successfully used to identify:

- Presence and content of oxidation in asphalt concrete;
- Type and content verification of asphalt additives including polymer modification and anti-stripping agent in asphalt concrete.

Clearly the literature offers positives, in that signs of binder ageing can be identified, and, in the case of the WRI study, spectroscopic results could be quantified and correlated to complex shear modulus G* to predict ageing. However, the most successful applications were performed in static mode; therefore the greatest challenge for development of the new tool would be its performance in motion. **Table 2** identifies some of the challenges and considerations which need to be addressed in the research project.

Table 2 Criteria for new tool

Criteria and technical challenges	Comments
Contact vs Contactless	• From the WRI research, it was found that a bitumen sample can be tested both in direct contact using transmission FTIR (T-FTIR) and photoacoustic (PA-FTIR). • Research should consider the use of ATR-FTIR or DR-FTIR.
Static vs Dynamic	• In both SHRP2 and WRI, testing was performed using static devices. • However, the aim of the research is to perform binder spectroscopy at traffic speed. • It is suggested that the assessment be initially performed on the static devices (e.g. handheld spectroscopy). • The assessment can then be carried out in motion (e.g. using handheld spectroscopy in motion).
Sensitivity (e.g. noise, vibration)	• The ideal device needs to perform under moving action. • Due to the lack of literature, it is suggested that the assessment is initially carried out in a controlled environment (e.g. handheld spectroscopy). • The device can then be assessed under various conditions (noise, vibration, etc.).
Failure identification	• Is it the ageing of the bitumen and/or; the change in the performance of the mixture as a whole?
Field contamination	• SHRP2 could not distinguish between the contaminations (e.g. motor oil, diesel fuel) using FTIR because of the overlapping bands of the two materials. • However, there is some evidence in the literature that contaminant residues in asphalt (decomposed tars and fuel) were effectively identified by FTIR [26]. • Further research should consider the effects of contamination.
Moisture, CO_2	• One of the challenges of field spectroscopy is that the presence of carbon dioxide could affect the spectrograph of the carbonyl absorption signature of an asphalt mixture. • However, both SHRP2 and WRI concluded that an alternative wavenumber (1200 cm^{-1}) has little atmospheric interferences.

In order to progress this research from feasibility into practice (i.e. Optical Spectroscopy of Binder Condition at Traffic Speed), the device needs to be able to function under moving conditions possibly under vibration and in the presence of contamination, moisture and CO_2.

The static (direct contact) ATR-FTIR method is not practical for use at traffic speed unless discreet (non-continuous) testing is carried out which involves sample capturing. This is proposed and discussed as a research option in **Section 8** (see **Figure 14**).

In order to allow continuous monitoring of the road surface at traffic speed, it is proposed that a non-contact DR-FTIR method is also explored. Currently there is no evidence suggesting its suitability for testing in motion, however, there is a possibility for expanding the current technology to allow continuous scanning by introducing an algorithm during data process. This will require new research to explore this possibility. The option for utilising this method is described further in **Section 8**.

8. Identify Programme of Research

Three main spectroscopic methods are proposed for testing optical spectroscopy of binder at traffic speed.

Concept 1: Sample capture and contact measurement using ATR-FTIR (see **Figure 14**).

In order to satisfy the research objective of testing the road surface at traffic speed, a concept has been developed that builds on existing sample capture technology using high pressure water jetting and vacuum sampling [4]. Bitumen coated samples recovered using this technique, typically have a size of 2/6mm. This concept allows analysis of the sample using ATR-FTIR which, from the literature, can be used to identify binder ageing; therefore a research programme could build on existing data.

In principle, this concept could test discreet samples (taken, for example at 1-5km intervals) which should be representative of the road section. If a sufficiently controlled and powerful enough single or group of jets could be developed to drill out mortar samples from the asphalt surface course at traffic speed then longitudinal strips of material could be vacuumed up and identified for later laboratory FTIR testing. This would reduce the need for complex monitoring equipment having to be on board the machine and would be effectively contactless (the jet and vacuum hose being above the surface). In addition, the contamination challenge in the FTIR (i.e. distinguishing between the contaminations because of the overlapping bands of the two materials) could be overcome by cleaning the asphalt surface, using the same equipment, prior to the sample collection. In principle, the sample recovery method can be applied without affecting the integrity of the asphalt surfacing.

Another advantage from this approach is that the collected samples can be subjected to further testing (e.g. in laboratory) and/or to be used for independent audit or calibrating the results from the mobile (on site) assessment. For example, conventional binder ageing rheological test methods could be used to test such samples. Furthermore, this technique would not have problems associated with environmental conditions. The samples could be dried as in the previous research programme [4].

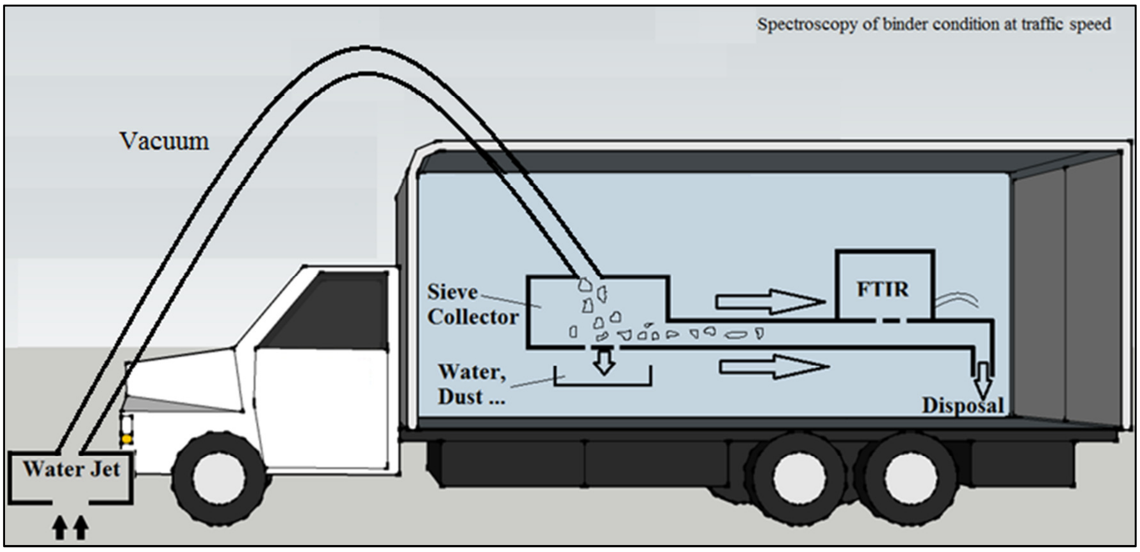

Figure 14 Sample capture and contact measurement using ATR-FTIR

Concept 2 Sample collection and testing using energy applied on the road surface

A second sample collection method is proposed that similarly involves sample collection prior to testing, possibly using heat or energy (laser) applied to the road surface with suction to collect gaseous samples (see **Figure 15**). This novel approach has not been considered in past work to our knowledge, and would require FTIR or other analytical equipment that is specific to analysis of gasses. Samples could be collected separately and tested in a laboratory so the machine need not carry the FTIR.

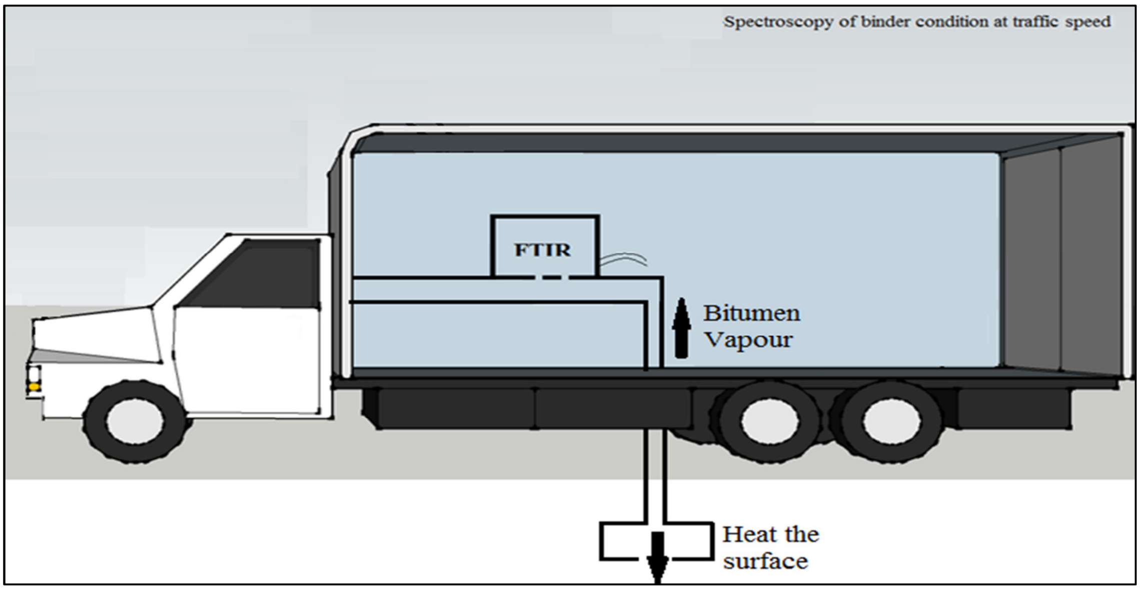

Figure 15 Sample collection and testing using energy applied on the road surface

Concept 3: Road surface analysis using DR-FTIR (non-contact method), see **Figure 16.** This concept involves direct, continuous measurement of the road surface.

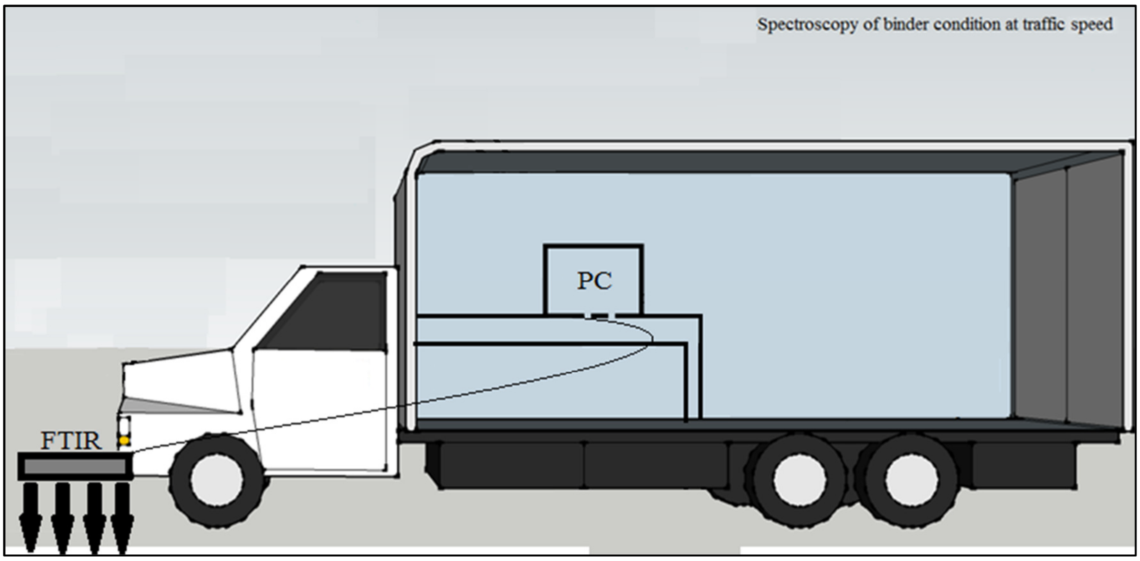

Figure 16 Direct road surface measurement using DR-FTIR

Clearly this concept has an advantage over Concept 1 in that it has the potential to allow continuous non-destructive measurement without additional sample processing steps. Whilst an initial study into this technique did not rule out the potential for use of DR-FTIR in motion, there are fundamental aspects that require research.

The scanning element alone takes around 10 seconds to scan through the range of 5000 to 650 cm^{-1}. There is scope to reduce the range of wavenumbers and speed up the test however, part of the spectrum that identifies contamination or other factors may be lost. To our knowledge testing in motion has not been done and therefore fundamental research is required.

A number of technical and practical considerations and challenges have been raised in **Table 2**. In order to identify a programme of research, the technical challenges have been further developed with the proposed concepts in mind. These challenges have been translated into objectives for further research, as summarised in **Table 3**.

Table 3 Objectives for Further Research

Criteria and technical challenges	Research Objectives
Achieving non-contact measurement and distance from sample	O1 Comparison of non-contact diffused reflectance FTIR (DR-FTIR) results to contact FTIR data. O2 Determine whether DR-FTIR can identify binder ageing. O3 Identify maximum distance between DR-FTIR source and sample (road surface).
Dynamic measurement (moving)	O4 Identify whether testing can be carried out in motion using DR-FTIR. O5 Determine the effect of motion on spectral resolution.
Speed of test	O6 Determine whether a shorter spectral window can be used to increase speed/resolution of test.
Effect of bitumen source and grade and age	O7 Identify and fingerprint a range of bitumen sources and grades. O8 Understand the effect of bitumen source, grade and age on spectral results.
Effect of bitumen additives (PMB, adhesion agents, fibres etc.)	O9 Identify and fingerprint common asphalt additives used in surface course. O9 Understand the effect of bitumen additives on spectral results.
Effect of contaminants at the road surface	Understand the effect of detritus, rubber deposits, etc. on the spectral results Decide whether to clean the surface before being tested
Effect of aggregate type	O10 Identify and fingerprint common aggregates. O11 Understand the effect of aggregate type on spectral results.
Effect of mixture type	O12 Understand the effect of asphalt mixture type on spectral results.
Threshold values related to ageing / failure	O13 Identify key trends / thresholds relating to binder ageing.
Sensitivity (e.g. noise, vibration)	O14 Understand the effect of noise and vibration on the spectroscopic equipment.
Effect of contamination on results (e.g. fuel, exhaust fumes etc.)	O15 Identify and fingerprint common contaminants. O16 Identify whether a cleaning / pre-treatment

	process to remove contamination is required.
Effect of moisture and atmospheric CO_2 on results	O17 Determine the effect of moisture and CO_2 on results.
Sample collection method	O18 Identify sample collection methods. O19 Determine the effect of the sampling method on the binder. O20 Determine whether the proposed method(s) of sample capture are practically and commercially viable.
Prototype development	O21 Develop vehicle mounted prototype for field testing. O22 Develop software programme for results processing.

9. Development of Research Programme

The research objectives identified in **Table 3** are not an exhaustive list. They form the basis for detailed development of a two stage research programme: fundamental and applied research (Stage 1), and product development (Stage 2).

Many of the research objectives presented in Table 4 can be considered fundamental research, i.e. the fundamentals of the spectroscopic techniques in relation to the materials and variables must be understood before applied research in the field can be fully implemented. Product development is also an important element of the research programme, which focusses on development of the vehicle mounted prototype equipment for end use. The proposed research programme is outlined in **Figure 17**.

The research programme outlined in **Figure 17** comprises the key research stages mapped over a five to six year period. Key decision gateways are included in this simplified model; however, due to the nature of the research, a number of iterations and feedback loops are expected.

The proposed research during year 1 focusses on the comparison of DR-FTIR with ATR-FTIR, and the feasibility of the use of DR-FTIR in motion. This research should aim to answer two fundamental questions;

1. Can DR-FTIR be used to indicate asphalt ageing?
2. Can DR-FTIR be used in motion?

If the answer to these questions is “no”, Concept 3 should not be pursued and further research should focus on sample collection and testing using ATR-FTIR. If the answer is “yes”, DR-FTIR should be prioritised, as Concept 3 best fulfils the brief.

Year 1 research should also consider the effect of contamination on results, along with the effect of varying bitumen grade, source and age, and begin to gather some site data.

Year 2 aims to build a library of spectral data to feed into future analysis software and considers the effect of asphalt additives and different aggregates used in asphalt surface course.

Years 3 comprise a programme of applied research which is more application focused.

Year 4-6 should focus on development of a prototype and software. In addition, the research project should include field trials to test the prototype within the programme period.

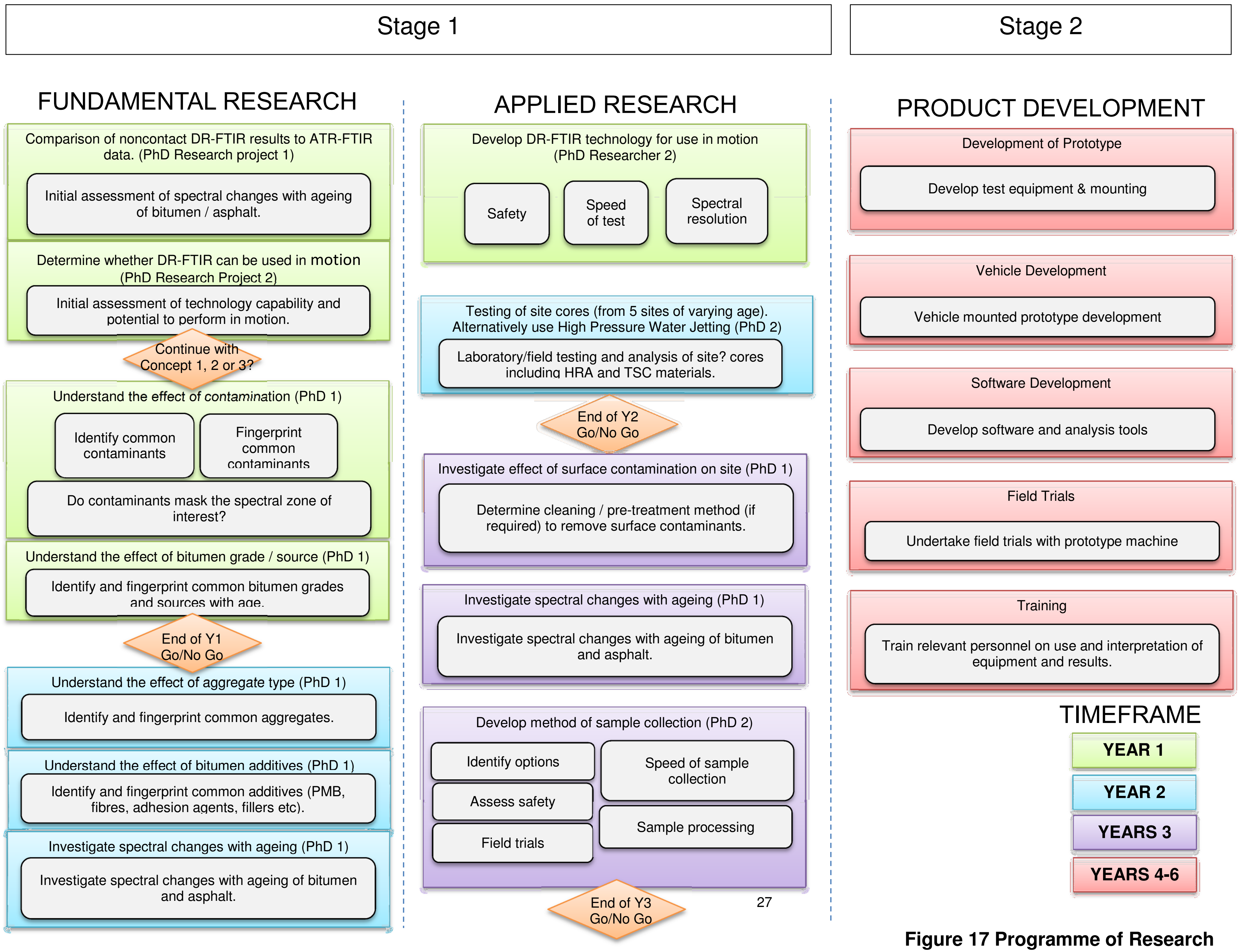

Figure 17 Programme of Research

10. Cost

The proposed research in **Figure 17** can be conducted in two stages as follows:

1) Stage 1 of the project will focus on the fundamental questions with the specific aim of addressing the research objectives presented in **Table 3**. It is suggested that universities are engaged by industry to appoint two researchers to potential PhD programs.

   One researcher will focus on addressing fundamental aspects, including:

   - establishing relationships between chemical composition of bitumen and/or asphalt with ageing,
   - measuring ageing by spectroscopy techniques
   - how results will be related to the overall performance of asphalt mixtures.

   The second researcher will focus on addressing the development of non-contact spectroscopy for use in motion.

   It is proposed for Stage 1 to be completed over 3 years as shown in **Figure 17.** There should be a number of go/no go decision gateways throughout Stage 1, with a major decision gateway prior to commencing Stage 2.

2) Stage 2 of the project focuses on product development, which includes prototype/ software development, vehicle mounting, training, and field testing. This stage spans 2 to 3 years as shown in **Figure 17.**

The total estimated budget for the stage 1 and 2 is estimated to be around £625,000 and £374,000, respectively, as detailed in **Table 4**.

Table 4 Budget estimate

	Description	**type**	**rate**	**quantity**	**total**
Year 1	Purchase FTIR + DH + DR	expenses	40000	1	40000
	Equipment setup	expenses	20000	1	20000
	sourcing material	labour	20000	1	20000
	Engineer/Researcher	labour	30000	2	60000
	Technician	labour	20000	1	20000
	Reviewer/Supervisor	labour	20000	1	20000
	PM & meetings @10% labour	labour	120000	10%	12000
	Sub-total= 192,000				
Year 2	**Description**	**type**	**rate**	**quantity**	**total**
	Field trials and testing	expenses	8000	10	80000
	sourcing material	labour	20000	1	20000
	Engineer/Researcher	labour	30000	2	60000

	Description	type	rate	quantity	total
	Technician	labour	20000	1	20000
	Reviewer/Supervisor	labour	20000	1	20000
	PM & meetings @10% labour	labour	120000	10%	12000
	Dissemination	expenses	3000	1	3000
	Sub-total= 215,000				
Year 3	**Description**	**type**	**rate**	**quantity**	**total**
	Field trials and testing	expenses	13000	5	65000
	Develop software - subs	expenses	40000	1	40000
	Engineer/Researcher	labour	30000	2	60000
	Technician	labour	20000	1	20000
	Reviewer/Supervisor	labour	20000	1	20000
	PM & meetings @10% labour	labour	100000	10%	10000
	Dissemination	expenses	3000	1	3000
	Sub-total= 218,000				
Year 4-6 *	**Description**	**type**	**rate**	**quantity**	**total**
	Field trials and testing	expenses	20000	5	100000
	Setup equipment	expenses	100000	1	100000
	Engineer/Researcher	labour	30000	1	90000
	Technician	labour	10000	1	30000
	Reviewer/Supervisor	labour	10000	1	30000
	PM & meetings @10% labour	labour	1500000	10%	15000
	Dissemination	expenses	9000	1	9000
	Sub-total=374,000				

Note: *budget estimate for 3 years.

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