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### ARTICLE INFO

## ABSTRACT

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Peel Durability Fracture Asphalt Asphalt road-pavements are sensitive to water ingress, which degrades the bitumen to aggregate adhesion, promoting failure. The effects of water on a range of asphalt systems have been quantified using peel tests. The bitumen binder on an aluminium backing was peeled from the aggregate fixed arm and the fracture energy was determined. In dry conditions, failure was cohesive within the bitumen, but became mainly interfacial between the bitumen and aggregate after immersion in water. The effect of water on the adhesion of bitumen to three aggregates (limestone, marble and granite) was evaluated. Acidic aggregates (granite) showed a greater loss of adhesion than basic aggregates (limestone and marble) under wet conditions. The porosity of the aggregates, although shown to be significant, was less important than their chemical composition. The interfacial adhesion in wet conditions can be improved by mixing a silane, amine or rubbery polymer into the bitumen.

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#### 1. Introduction

Asphalt mixtures, consisting of mineral aggregates bound with a bitumen binder [1], are used extensively as road surface materials. Although asphalt is a relatively cheap material [1], the disruption to traffic flows and costs of replacing degraded road surfaces are significant, leading to a demand for more durable materials. Water is a major cause of such premature failure in asphalt. The resulting water damage causes a loss of stiffness and structural strength, due to the loss of adhesion between the aggregate and the bitumen, and/or loss of the cohesion within the bitumen binder [2–4]. Hence, an understanding of the adhesion mechanisms between the aggregate and bitumen is required if the durability performance of road surfaces are to be improved and an optimum selection of the asphalt component materials are to be made.

The effects of water on asphalt mixtures have been studied extensively. Both experimental and computational methods have been developed to assess their durability and their response to water ingress [4–12]. The experimental methods include qualitative tests conducted on loose bitumen-coated aggregate, such as the boiling test [7], and quantitative tests conducted on compacted asphalt mixtures [8], such as the Saturation Ageing Tensile Stiffness (SATS) test [10,11]. These approaches are frequently complex and not sufficiently sensitive to discriminate between the

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performance of different types of bitumen binder and aggregates, and hence cannot give specific information on the nature of the bitumen–aggregate interface. Computational approaches have been developed to simulate the in-service conditions experienced by asphalt mixtures, and hence to predict their durability and water-resistance [5,6,13,14]. However, due to the lack of understanding of the adhesion mechanisms between the bitumen binder and the aggregates, and how such interactions are affected by the presence of water, these methods do not generally provide definitive guidance for selecting asphalt mixtures or for quantifying the improvement in performance from the addition of adhesion promoters.

Recently the present authors showed that a fracture mechanics approach can overcome these problems, and that such an approach can be used to quantify the effect of water damage in asphalt [15]. The use of the peel test [16–18] can avoid many of the problems associated with the viscoelastic nature of bitumen. The peel test allows the measurement of the adhesion between the bitumen and the aggregate (i.e. the adhesive fracture energy) and it has been adapted such that the water-resistance of different bitumen–aggregate combinations can be assessed following immersion in water for several days. By measurement of the fracture energy, the durability of bitumen–aggregate joints can be quantified [15]. This approach also provides information on the fracture path and evaluates the adhesive and/or cohesive strength of the joint.

Previous studies have indicated that the susceptibility of asphalt mixtures to attack by water is related to the mineralogy

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and surface texture of the aggregate, and also to the adhesion between the bitumen binder and the aggregates [1,4,19–21]. Airey and co-workers [4,12] assessed the water-damage of asphalt mixtures by comparing the stiffness of unconditioned and waterimmersed specimens. It was found that significantly less waterdamage occurred when basic aggregates, e.g. limestone, were used in the mixture than when acidic aggregates, e.g. granite [4] were used. In an attempt to explain this observation, both the physical and chemical properties of the aggregates were studied. Abo-Qudais and Al-Shweily [19] showed that a limestone aggregate had greater resistance to water-damage than basalt, and explained that limestone is positively charged, leading to stronger bonds, and as a result is a hydrophobic aggregate. They suggested that the chemistry of the aggregate affects the degree of water sensitivity of the bitumen-aggregate bond and noted that silica usually causes a reduction in bond strength between bitumen and aggregate; as the limestone aggregate contains less SiO<sub>2</sub> than basalt it shows a better resistance to water. Another study using granite aggregates also showed that the mineralogy of the aggregates has a significant impact on their adhesion to bitumen [20].

It is clear from the literature that the durability of asphalt mixtures (and hence the service life of road surfaces) depends, at least in part, on the adhesion between the bitumen and the mineral aggregates. In practice, the selection of the bitumen binder and aggregate during road construction is governed largely by economics: the cost of transporting the heavy aggregates any significant distance is prohibitive so the aggregates are sourced locally to the road construction site. Thus, the aggregates used on road surfaces reflect the local geology. For this reason there are wide variations in the durability of asphalt mixtures and various methods have been employed to improve them. For example, several methods have been used to reduce the extent of debonding (also known as stripping), including the addition of fillers, of polymers and of amine anti-stripping agents [22,23]. Also, organosilanes have been successfully used to prevent stripping of asphalt mixtures [23-25].

In the present work a fracture mechanics approach has been followed to quantify the adhesion between the bitumen binder and the aggregate in selected asphalt mixtures. The fracture mechanics parameter,  $G_{A_1}$  (or fracture energy) reflects both the energy required to break the intrinsic molecular forces associated with interfacial or cohesive failures and also the energy dissipated locally in the plastic or viscoelastic process zone at the crack tip. Attempts to improve the fracture energy therefore either work by increasing the intrinsic adhesion or by increasing the locally dissipated energy in the bitumen. The first objective of the work is to use the fracture mechanics approach to quantify the relationship between the water-damage performances of the asphalt mixtures as a function of the aggregates used. The second



Fig. 1. Images of the four aggregates.

objective is to quantify the extent to which the water-damage performance can be improved by the use of various additives including silane and amine-based adhesion promoters and also the use of a polymer-modified bitumen.

### 2. Experimental

The peel test has been used in this work due to the viscoelastic and relatively low-modulus characteristics of the bitumen binder [15]. In this section, first the constituents of the asphalt mixtures are described and then the details of the adhesion promoters used are presented. Second, the experimental techniques employed including the peel test, the water exposure and the aggregate water uptake studies are presented.

#### 2.1. Materials

The same grade of bitumen binder was used throughout this work and it was a medium penetration, 40/60 pen, provided by Shell Bitumen (Manchester). (The 'penetration' number is defined as the distance, expressed in tenths of a millimetre, travelled by a needle into the bitumen under a known load, at a known temperature for a known time [1].) Four different aggregates, each possessing a different chemical composition and porosity were studied, as shown in Fig. 1. Two basic aggregates, limestone and marble, and two acidic granite aggregates were chosen for study. Limestone has a relatively good resistance to water [1,4] and was therefore selected as the standard aggregate (for use in the control tests). Marble has a similar chemical composition to limestone but is less porous, and was selected so that the effect of aggregate porosity on the resistance to water could be studied. Limestone is a sedimentary rock and is composed of calcium carbonate fossils, while marble is recrystallised into interlocking calcite crystals [26]. The two granites were selected as they are reported to impart poor resistance to water to the asphalt mixture [20]. The chemical compositions of the aggregates, analysed using mineralogy analysis (MLA) by the University of Nottingham, are summarised in Table 1. (MLA uses backscattered electron (BSE) and energy dispersive X-ray (EDX) signals obtained using scanning electron microscopy to determine mineral composition. Comparison with a database of minerals and image processing allows particle boundaries and minerals to be identified.)

Three strategies to improve resistance to water, namely the use of silanes, amine anti-stripping agents and polymer modifiers were compared. The two silanes selected were supplied by Sigma Aldrich. The first was trimethoxy(octyl)silane (TMOS) which has a short carbon chain plus the silane functional group. The second silane was 3-(2-aminoethylamino) propyltrimethoxysilane (APT-MOS) which has two additional amino-functional groups. The silanes were mixed individually into the bitumen at a ratio of 0.5% v/v. In addition, a commercial amine-based anti-stripping agent (ABAA) was used, supplied by the University of Nottingham. Finally, a polymer modifier was used and this was a styrenebutadiene-styrene (SBS) copolymer, supplied also by the University of Nottingham. The anti-stripping agent and the polymer modifier were directly mixed into the heated bitumen prior to making the peel test specimens. The materials used are summarised in Table 1, where the silica and carbonate contents are given.

#### 2.2. Peel test description and procedure

In the peel test, a flexible arm (the peel arm) is bonded to a rigid substrate (the fixed arm) with an adhesive [15,17]. The flexible arm is then peeled from the fixed arm and the peel force

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