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# Surface analyses of bitumen in a context of decarbonation: importance to introduce low frequency dielectric spectroscopy investigation

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## ABSTRACT

Non-destructive methods by ground-penetrating radar (GPR) are widely used for road evaluation. To analyze the propagation of electromagnetic waves, it is necessary to have information about the dielectric properties of roads. However, multi-layered structures, various constituents, and a large scale of roads make the direct measurement of core samples hard to be representative. Moreover, climatic conditions will also complicate the analysis due to the dielectric drift. Thus, a non-destructive dielectric measurement adapted for road *in situ* study is very important. In this work, we will present an open-ended coaxial probe and its application on a newly paved road. In the future, we will integrate the impedance analysis at low frequency to test the dielectric relaxations in different controllable environments, which will help us to better understand the dielectric properties of roads as well as the influences of climate changes.

**Keywords:** dielectric permittivity, open-ended coaxial probe, road pavements

## 1. INTRODUCTION

Surface diagnostics conducted to assess the health of roads and prevent hazards and other disasters call for various non-invasive techniques: ground-penetrating radar (GPR) is one of the most effective tools to evaluate the geometry of roads <sup>[1]</sup>. GPR auscultates the travel time of electromagnetic (EM) waves in a material, known as the temporal signal. The travel velocity of EM waves is governed by the road material's dielectric permittivity. Interpreting the temporal signal and evaluating the geometry of the road require precise knowledge of the dielectric properties of different materials in the roads. Hence, characterizing dielectric properties of road materials is essential for the road's diagnosis by GPR.

The dielectric properties of a road depend on several factors including materials (mineralogical types of aggregates, compactness of layer, grade of bitumen, etc.), and environments (temperature and humidity, etc.). Direct measuring the road's dielectric constant by taking off the core samples in the laboratory is a common way, but the token off core samples need to have rigid dimensional requirements for adapting the device under test (DUT) in the coaxial transmission/reflection cells<sup>[2, 3]</sup>. Moreover, the exact microwave dielectric characterization for several core samples is hard to be representative for the whole road due to the heterogeneous structures. These bring inconvenience for road dielectric characterizations. Another common way to estimate a road's dielectric constant is to apply mixing laws <sup>[4]</sup> based on the volumetric fractions and dielectric constants of the constituents. However, these values are not always easily accessible, especially, recently, the application

of modified bitumen and recycled materials have been applied in the road's construction to achieve reducing carbon emissions. Take bitumen as an example: there are different grades in conventional bitumen. The dielectric properties of these conventional bitumen of different grades do not have an obvious difference (2.4 - 2.6) <sup>[3]</sup>. Thus, it does not obviously affect the dielectric properties of road materials since the bitumen rate is low compared with the rock proportion. However, the modified bitumen is not in this case. These modifiers can be biofuels (organic plants, animal corps, byproducts of forest or agriculture), additives (emulsions, resin, polymer, crumb rubber), recycled materials (recycled bitumen, cooking oil, waste engine oil), etc. <sup>[5]</sup>. The source of modifiers is extremely large with different physicochemical properties, which can largely affect the dielectric properties of modified binders. Meanwhile, the interaction between the binder and aggregates will also alter due to the different physicochemical properties of non-conventional materials. Both will make the road dielectric permittivity more frequency-dependent. There are lots of publications in bitumen and its modifiers (about 6500 in Web of Science including review papers before 08/2022), most of them focus on the rheology properties, but if we refine the research field by dielectric and electromagnetic properties, the number decreases sharply to 65, and most of them focus on conventional bitumen and oil sands. Less than 10 papers have focused on the modified bitumen's dielectric properties <sup>[6, 7]</sup>. Since either the direct measurement of core samples or the application of mixing laws has their limits, developing an adaptable and *in situ* dielectric characterization of roads will be useful.

In microwave domain, dielectric tests by open-ended probes are attractive for road characterization because of their non-destructive characteristics <sup>[8, 9]</sup>. Thus, in this work, we will introduce an on-site application of an open-ended coaxial probe for road pavement. The work will be organized into two parts: the first part is the laboratory measurement for known homogeneous polyvinyl chloride (PVC) samples to evaluate the open-ended test. The second part is an on-site investigation of a newly paved road in France. The open-ended coaxial probe will be used to map the dielectric constants of the surface course on a sunny day and a rainy day, respectively. A point of awareness regarding the benefits of low-frequency dielectric testing in the context of future new bitumen will conclude the presentation of our work.

## 2. LABORATORY MEASUREMENTS

The dielectric constants of an assemblage of PVC layers (each square layer: thickness = 5 mm; surface = 400 cm<sup>2</sup>) and PVC cylinders (diameter = 75 mm; height = 70 mm) were tested by the same coaxial line but under two configurations: a reflection/transmission cell configuration and an open-ended configuration as presented in Fig. 1 (a). The cell configuration is a validated method <sup>[3]</sup> and the tested permittivity ( $\epsilon_r$ ) of the PVC cylinders is 2.9-0.03j from 50 MHz to 1.1 GHz. We will use this value as a reference to evaluate the open-ended results. For the open-ended test, the calibration is based on the capacitance approach <sup>[8, 9]</sup>. The cut-off frequency depends on the aperture of the probe (inner diameter = 32.2 mm; outer diameter = 75 mm), as well as the dielectric constant of the sample. Herein, the cut-off frequency is not over 0.7 GHz for  $\epsilon_r= 2$ , and 0.4 GHz for  $\epsilon_r= 5$ .

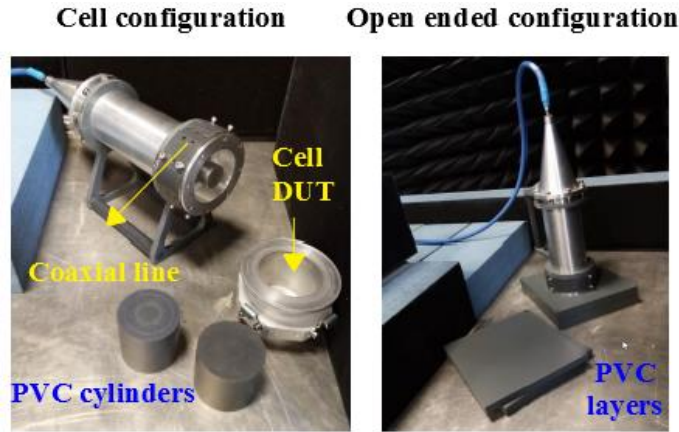


Figure 1 Dielectric measurement by two configurations

The dielectric constant of PVC assemblage tested by the open-ended probe is presented in Fig. 2 (a) and (b). When the layer number is less than 3 (i.e. thickness = 1.5 cm), the dielectric constant of PVC is higher than the cell value, and with obvious resonant peaks in the imaginary part from 0.2 to 0.3 GHz in Fig. 1 (b). Only when the layer number is over 8 (i.e., thickness = 4 cm), the resonant peak in the imaginary part disappears. Meanwhile, the dielectric constant starts to be independent of the layer numbers and approaches the cell value. These results indicate a minimum thickness ( $t_{\min}$ ) when using the open-ended probe. Theoretically,  $t_{\min}$  depends on the aperture of the coaxial line, i.e.  $t_{\min} >$  radius of the hollow external cylinder. Herein, the radius is 37.5 mm which makes the  $t_{\min}$  of the PVC should be over 4 cm. This is agreeable with the tested PVC assemblage (at least 8 layers). On the other hand, the dielectric constant of PVC assemblage with 20 layers obtained by open-ended configuration is from 2.63-0.12j to 2.57-0.04j when the frequency is from 50 MHz to 0.7 GHz, where the real part is a bit lower than the cell value (2.96-0.03j). One possible reason comes from the limited sample scale as an open probe assumption is to have a large size considered infinite. This is difficult to achieve for laboratory measurement, but for on-site road investigation, this hypothesis can be better adapted.

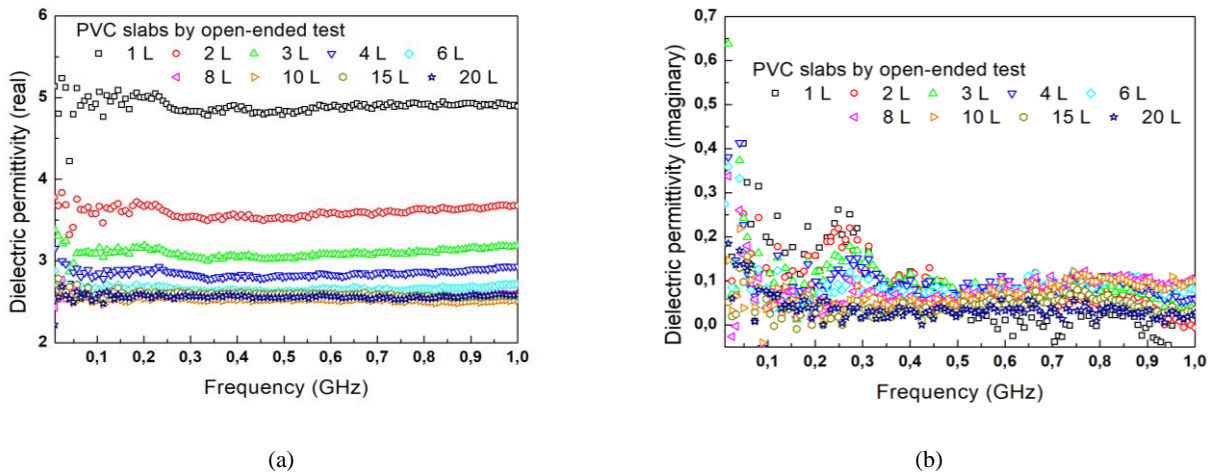


Figure 2 Dielectric permittivity of PVC slabs with different layers (L) tested by open-ended probe: (a) for the real part and (b) for the imaginary part.

### 3. ON-SITE INVESTIGATION

Roads are with different layers arranged in the order of descending load-bearing capacity: i.e., surface course, base course and subbase course, as presented in Fig.3. The nomenclature of the layers is as follows:

- BBSG: binder course made by semi-coarse hot-mix asphalt (an average thickness of 5 cm);
- GB: base course made by bituminous gravel (an average thickness of 18-20 cm);
- GNT: subbase course without bitumen.

Surface course is the most expensive layer constructed by bitumen in which it also has a layer-by-layer structure as indicated in the three core samples taken from the tested site, located in Pays de la Loire, France. Because of this layer-by-layer structure, roads need regular controls to ensure resistance to cyclic loading. Generally, the dielectric constant of bitumen based surface course is around 4. Thus, considering the cut-off frequency of the open-ended probe due to its geometry (from 50 MHz to 0.5 GHz when dielectric constant of the sample is about 4), we use the dielectric constant at 0.35 GHz for the discussion.

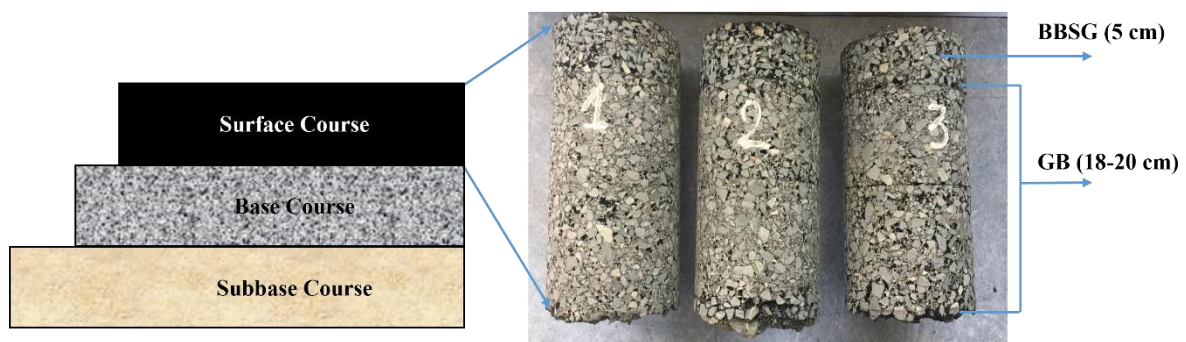


Figure 3 Schematic of a road with different layers and the photos of the core samples taken off from the AXE zone in the tested site

The tested field is illustrated in Fig.4. It has three zones based on the function of circulation from the center to the board: central stripe (AXE), straight tread (BRD) and emergency stopping lane (BAU), where AXE and BRD have two layers (BBSG and GB) paved on the subbase course (GNT); BAU has only BBSG layer paved on GNT. Our investigation was conducted on three profiles positioned in BAU, BRD, and AXE zones, respectively. Each profile had 21 points with intervals of 10 m.

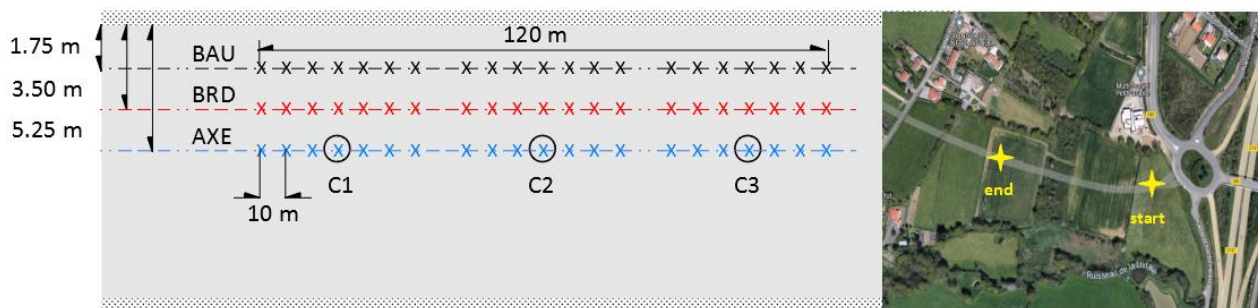


Figure 4. Schematic of the tested site and its satellite map. C1, C2, and C3 were the positions of the core samples in the AXE zone

The construction information of the surface course was provided by the Agence d'Angers of Cerema - Groupe Infrastructures Durables. The mineralogical type of the aggregates in BBSG and GB is micro-diorite with a dielectric constant of about 5.3 [10]. The content of the bitumen (35/50 penetration grade, with the dielectric constant of 2.5) is 5.1% in BBSG, and 4.5% in GB. Three core samples were taken out on AXE profile for the gamma-densitometer test to have the compactness of BBSG and GB [11]. The values of compactness are listed in Table 1.

Table 1. Compactness of BBSG and GB layers in the three core samples tested by gamma-densitometer.

	<b>C1</b>	<b>C2</b>	<b>C3</b>
<b>BBSG</b>	87.1 %	81.3 %	84.8 %
<b>GB</b>	94.3 %	93.9 %	92.8 %

Different mixing laws in Ref 4 including Maxwell Garnett (MG), Inverse Maxwell Garnett (IMG), Coherent Potential (CP), Bruggeman Symmetric (BG), Lichtenecker (LI), Looyenga-Landau-Lifshitz (LLL) and Complex Refractive Index Model (CRIM) are used to calculate the dielectric constants of the three core samples based on the volumetric fractions and dielectric constants of air, bitumen, and aggregates. The results are presented in Fig.4 (a)-(c). The measured dielectric constants of the core samples are in the range of the calculated results between BBSG and GB, especially for the range proposed by Maxwell Garnett (MG) model. This agrees with the assumption that the dielectric constant derived from the open-ended probe is a global value of the surface course. Since the calculation of the mixing law agrees with the dielectric values tested by the open-ended probe in the core sample regions, a dielectric map at 0.35 GHz of the three profiles (BAU, BRD, and AXE) on a sunny day (March 29, 2022) and on a rainy day (April 5, 2022) are plotted in Fig. 5. To remind, the road was paved on March 28 (2022) and the test was on the next day. On April 5 (a rainy day), the climate was humid with a small rain, not a deluge. There was no circulation during the whole test period.

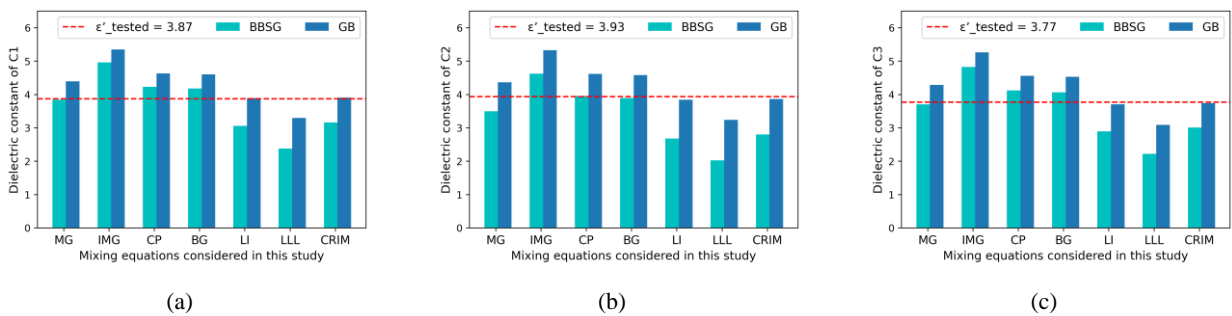


Figure 5. Dielectric constants calculated by mixing equations for three core samples: (a) for C1, (b) for C2, and (c) for C3. Mixing laws used in the calculation were listed in Ref 4: Maxwell Garnett (MG), Inverse Maxwell Garnett (IMG), Coherent Potential (CP), Bruggeman Symmetric (BG), Lichtenecker (LI), Looyenga-Landau-Lifshitz (LLL) and Complex Refractive Index Model (CRIM)

As indicated in Fig 5 (a), some variety can be found among the tested points on a sunny day. The variety is related not only to the heterogeneous structure but also to the texture of the road, because the surface roughness will change the effective contact between the sample and the coaxial probe. Furthermore, the dielectric differences between sunny and rainy days are not only related to the humidity, but also to the surface texture: It is known that the water is with a large dielectric constant of 80, which is probably the main reason for the

increased dielectric constant under a rainy day. However, as a newly paved road without circulation, there are little fissures on the surface, where the water is hard to penetrate the surface course mixed with bitumen. Thus, the water mainly stays on the road's surface, but the unsmooth surface affects the water accumulation on the road, which will bring the dielectric difference in Fig.5 (b). Although we have tested the road's dielectric constants on a rainy day, at this moment, we cannot do a humidity estimation based on the dielectric constant since we do not have the texture analysis. We will complete this part by laser scanning in the future. In addition, limited by the cut-off frequency of the open-ended probe due to the geometry of the coaxial line and the dielectric properties of roads, the frequency is from 1 MHz to 0.4 GHz, which may be not large enough for applying dielectric relaxation models to understand the evaluation of microstructure caused by environmental factors. Hence, it will be useful to measure the dielectric properties at low frequency by impedance analysis in a controllable environment and the texture laser scanning, both will be useful for us to understand the dielectric properties of roads under different climate environments.

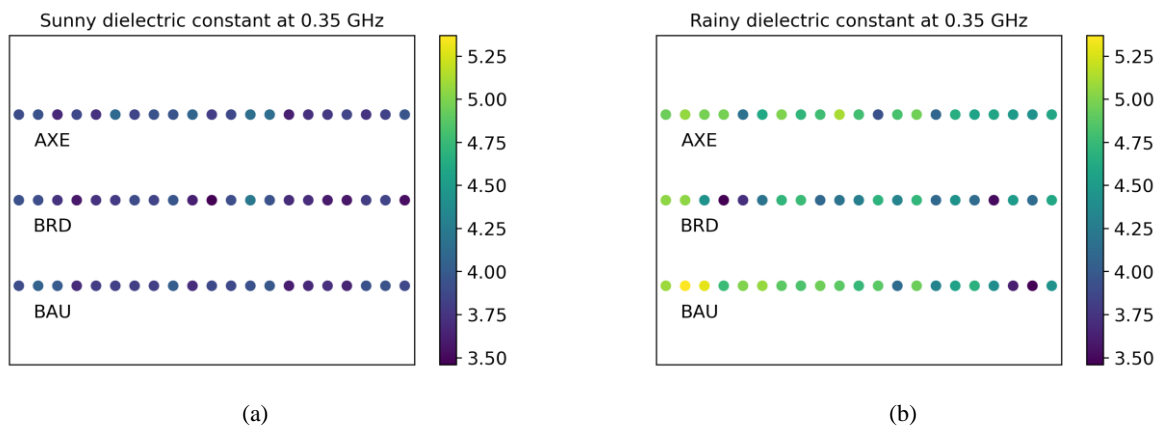


Figure 6. Color map of the dielectric constants tested at 0.35 GHz for three profiles under a sunny day and a rainy day

In addition, the emerging application of non-conventional road materials will increase the frequency dispersion, which will make the climatic influences more complicated to diagnose. Thus, a study of dielectric properties in the microwave domain will no longer be sufficient to provide a non-invasive diagnostic of road pavement conditions, and in particular, of the degradation of the underlay. In the future, we will integrate low-frequency impedance analysis to study dielectric relaxations of heterogeneous structures in different controllable environments (moisture, heat, and UV), which will help us expand the frequency range tested and understand the dielectric properties of roads as well as the influences of climate change in a more comprehensive way.

#### 4. CONCLUSIONS

A non-destructive dielectric measurement by an open-ended probe has been discussed in two cases: a laboratory test and an on-site investigation. In the laboratory part, the dielectric constant of PVC slabs tested by the open-ended probe has been found to be a bit lower than that tested by the validated test due to the limited scale, but this effect should be lower in the on-site investigation because of the infinitive scale of the road. Thus, in the on-site part, we can test the dielectric constant of a newly paved road on a sunny day. The obtained dielectric constant of the road agrees with the calculated value by mixing laws based on the compactness tested by

gamma-densitometry, which infers the open-ended probe can be adapted for road's in situ dielectric measurement.

Although we have also tested the road's dielectric constants on a rainy day, in this work, we cannot do a humidity estimation based on the dielectric constant since we do not have texture scanning by laser. We will complete this part in the future. In addition, limited by the cut-off frequency of the open-ended probe, the frequency is from 50 MHz to 0.4 GHz, which may be not large enough for applying dielectric relaxation models to understand dielectric drift. Hence, the measurements under controllable environments (including humidity, heat, and UV) by impedance analysis at low frequencies can be very useful. We will continue this in the future for a better understanding of environmental factors on road dielectric properties.

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