Measurement System for Evaluating Dielectric Permittivity of Granular Materials in the 1.7–2.6-GHz Band

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Abstract—The design and the experimental characterization of a waveguide system for complex permittivity measurements on both solid and granular materials are presented. The proposed system is intended for the dielectric characterization of asphalt concrete and of its components at frequencies around 2.45 GHz. Therefore, the system provides measurements on granular materials, such as mineral aggregates found in the asphalt concrete. A series of experimental tests on reference materials shows, via comparison with a standard measurement system, errors within 1%. A theoretical analysis of uncertainty contributions confirms a predicted expanded uncertainty lower than 2%. Furthermore, to test the proposed system for dielectric spectroscopy on materials typical of the asphalt industry, measurements were also performed on calcareous and basaltic materials, which are typically used for the production of asphalt. Finally, three different dielectric models (namely, α model, Ansell’s model, and Topp’s equation) were comparatively assessed to identify the most suitable ones in describing the water content and dielectric permittivity relationship for the considered materials.

Index Terms—Asphalt concrete characterization, permittivity measurements, transmission/reflection methods, uncertainty evaluation, WR430 waveguide.

I. INTRODUCTION

The development of dielectric spectroscopy techniques, with particular emphasis on those operating in the microwave frequency range, has attracted an increasing interest in the last few years [1]. Such techniques permit measurement of the frequency-dependent complex permittivity of a material sample. Knowledge of this quantity can prove useful for quality-control purposes [2], [3] or for understanding the interaction of the material with an electromagnetic wave and the corresponding heating effect [4]. A specific field of application of microwave heating, especially at the 2.45-GHz ISM frequency, is road maintenance [5]. Indeed, by heating the asphalt concrete through the use of microwaves, it is possible to perform on-site repairs of holes or cracks present on the road surface. Still, microwaves might even be employed in the production stage of the asphalt concrete, as a substitute to conventional ovens currently exploited for drying and heating the mineral aggregates, which are subsequently bound together with asphalt. Microwave heating might also help for the recycling process of reclaimed asphalt pavement.

Mineral aggregates used inside the asphalt concrete mixture have different sizes, typically ranging from less than 1 up to 25 mm in diameter. They are obtained from basalt or calcareous rocks. In the final concrete, they are bound together by asphalt (or bitumen), which is usually refined from petroleum. The key data needed for studying the feasibility of a microwave system for asphalt concrete production or recycling are the complex permittivities of the concrete components (asphalt and mineral aggregates) and the concrete itself. Therefore, an accurate system suitable to perform complex permittivity measurements around 2.45 GHz on both compact and granular materials is needed. The most common solutions in this frequency range exploit open-ended coaxial probes [6], whose tip is placed in contact with the material [7]. However, such systems are not suitable to perform measurements on granular materials and have a sensitive area that is definitely too small to allow a correct characterization of asphalt concrete, which is a mixture whose granular components can also be several tens of millimeters in diameter. To overcome such limitation, transmission/reflection solutions can be adopted, where the material is placed inside a suitable section of transmission line [8]. However, currently, implemented systems are based on a coaxial structure [9], [10] and operate in a frequency region well below the ISM band at 2.45 GHz. On the other hand, the most widespread waveguide measurement systems are based on the WR90 standard waveguide, allowing measurements in the 8–12-GHz band (X-band). In this paper, the design and the metrological characterization of a waveguide system, suitable to perform complex permittivity measurements on material samples of...
adequate size, are presented. The system is developed with the aim of providing an accurate and affordable solution for measurement on granular and compact materials in the frequency range around 2.45 GHz.

To test the performance of the proposed system, measurements were also carried out on typical materials used for asphalt, moistened at different water content levels. Finally, different dielectric mixing models were investigated and their suitability for the considered materials was verified.

II. DESIGN OF THE WAVEGUIDE SYSTEM

As reported in [11], the design goal of achieving a permittivity measurement system suitable to characterize adequately sized material samples (possibly granular), suggested the use of a transmission/reflection system based on rectangular waveguides.

Among the different standardized waveguides, the WR430 waveguide, spanning the 1.7–2.6-GHz region, was used. The choice of this waveguide over other standard waveguides with partially overlapping operating frequency range (i.e., the WR340) was due to its larger cross-sectional dimensions (approximately 109 mm × 55 mm), thus accommodating samples with sufficient volume to accurately characterize the asphalt concrete mixture and components.

The whole system was designed through electromagnetic full-wave simulations, performed employing CST Microwave Studio software. A sketch of the designed system is shown in Fig. 1(a). The system is composed of a couple of coaxial (n-type) to rectangular waveguide (WR430) transitions; two standard WR430 waveguide sections (long enough to ensure that higher order modes are sufficiently damped); and a sample holder, 10 cm in length.

Typically, waveguide systems are employed for measurements on solid and compact materials, which can be cut in a parallelepiped shape and inserted in the sample holder. However, one of the goals of the proposed measurement system is the characterization of granular materials (such as the mineral aggregates). Therefore, to allow the insertion of granular samples in the sample holder, two pressurization windows were added, directly connected to the sample holder ends, which are filled by epoxy and confine the material inside the holder. The windows are designed so as to bear up to 1 kg of material. Due to the presence of the epoxy dielectric, a shunt capacitive load is added to the waveguide, thus creating an impedance-mismatch effect. Even though this can be compensated for through standard vector error correction procedures, the windows were equipped with a printed inductive/copper iris. Such an iris, typically realized through two thin rectangular metal patches protruding from the narrow walls of the waveguide (so as to reduce its effective width), is designed to provide a shunt inductance able to cancel, through resonance, the capacitive mismatch at the target frequency of 2.45 GHz [12]. To slightly enlarge the matching bandwidth, the iris was designed with an elliptically shaped (rather than straight) contour, whose eccentricity was optimized through specific electromagnetic simulations. The whole structure is made of brass, with copper-plated inner walls.

To make the system practically usable for performing reflection/transmission measurements with a vector network analyzer, it is necessary to apply the vector error correction procedure at the sample holder ports [13]. Such procedure permits compensation for most of the systematic errors and requires a specific calibration kit. The most simple calibration standards to be employed for waveguide systems are the short circuit (i.e., a metallic end plate) and a waveguide section of known length (typically equal to one-fourth of the wavelength computed in the middle of the operating frequency band). This set of standards accommodates the thru-reflect-line (TRL) vector error correction procedure. Unfortunately, there are several waveguide calibration kits available on the market; however, to the best of authors’ knowledge, none of them is suitable for the WR430 waveguide format. Therefore, the short circuit and waveguide section standards were built as part of the whole project. Starting from their geometrical and electrical characterization, a custom model for the developed calibration kit was implemented and added to the network analyzer database [14].

III. MEASUREMENT MODEL AND UNCERTAINTY

In transmission/reflection measurements, the sample is placed inside the waveguide and an electromagnetic wave is launched at the waveguide port. By measuring the scattering parameters at the waveguide ports, it is possible to retrieve the complex permittivity of the sample through an appropriate measurement model.
Historically, the first works that proposed permittivity measurements with the transmission/reflection technique date back to the mid-20th century [15]–[17]. However, it was only in the early 1970s that a new model was developed in [18] and further optimized in [19]: such model, known as Nicolson–Ross–Weir (NRW), accurately operates only at frequencies far from the sample resonances, which occur whenever the sample length becomes an integer multiple of half wavelength computed inside the sample.

An alternative model for evaluating the complex permittivity of the sample, starting from the measured scattering parameters, has been developed by the National Institute of Standards and Technology (NIST) [8], [20]: such a model employs a mathematical formulation that largely limits errors related to sample resonances.

In this paper, measurements were performed using an Agilent E8363C vector network analyzer, equipped with Agilent 85071E permittivity measurement software [21]. In particular, this software provides different measurement models, including the aforementioned NRW and NIST models. The measurement results reported herein have been obtained employing the NIST model, which has been proved to be the most accurate for nonmagnetic materials, like the ones of interest for this paper.

A. Uncertainty Contributions

A theoretical evaluation of permittivity measurement uncertainty for the NIST model can be found in [8] and [20]. Applying such evaluation to the proposed system, the following main uncertainty contributions can be identified: 1) uncertainty in scattering parameters; 2) air gaps between sample and holder; 3) dimensional uncertainty in the sample holder; 4) uncertainty in sample length; and 5) uncertainty in the exact sample positioning inside the holder.

In measurements on solid and compact materials, many of these contributions were made negligible thanks to the chosen mechanical tolerances in the system realization and to the accurate milling of the material samples. Therefore, the main remaining contributions are likely those related to scattering parameters and to the sample length. In particular, the uncertainty in sample length is mainly the result of the imperfect planarity of the sample surface, especially for pliable materials.

The exploiting results reported in [8] for the sensitivity coefficients, using uncertainty figures taken from the network analyzer datasheets for scattering parameter measurements, and assuming a worst case uncertainty of 0.05 mm in sample length (on the basis of the measured nonplanarity), the combined uncertainty on the real part of the measured permittivity is about 2% (average value over the entire frequency band). This result is in optimum agreement with typical uncertainty stated in the 85071E software specifications [21].

Clearly, for measurements on granular materials, some of the aforementioned uncertainty contributions may become more prominent. For example, the sample length is harder to be accurately evaluated. In addition, inhomogeneities in the sample may lead to the propagation of higher order modes. Higher order mode propagation, in particular, is responsible for a deterioration in the accuracy of scattering parameter measurements, which become affected by spurious oscillations. These artifacts on the scattering parameters can lead to an increased uncertainty in estimated permittivity, especially around sample resonances, where the sensitivity coefficients for the NIST model tend to become rather large. However, as long as an average value of the permittivity is of interest, because no significant relaxation processes are expected in the measurement bandwidth, artifacts around sample resonances are not of great concern and should not decrease measurement accuracy in a significant way.

IV. EXPERIMENTAL CHARACTERIZATION OF THE PROPOSED SYSTEM

To characterize the presented WR430 custom system, measurements were performed on a set of different solid materials. Unfortunately, while for reference liquid materials, very accurate databases of permittivity are available [22], it is more complex to obtain reliable reference solid materials. Therefore, we used low-permittivity solid materials that are known to exhibit an extremely flat permittivity over the entire microwave frequency band. In this way, it was possible to validate the system through a comparison with measurements performed on the same materials employing a standard WR90 waveguide system operating between 8.2 and 12.4 GHz, and used as a reference. Indeed, the WR90 waveguide system is entirely based on commercial components (with the exception of the sample holder, realized with a milling machine with 0.005 mm repeatability), and it is an excellent candidate to be used as a reference for comparing the WR430 custom system measurements, thanks to its stated typical worst case uncertainty equal to 2% [21].

For the characterization of the system, the following materials were chosen: 1) low-density polyvinyl chloride; 2) polytetrafluoroethylene; 3) polymethyl methacrylate; and 4) polycarbonate. These test materials were chosen to satisfy the following criteria:

1) flat permittivity spectrum so as to allow a direct comparison between the results obtained with the WR430 and the WR90 systems;
2) low permittivity (real part less than 8), in order to better represent permittivities of asphalt concrete main components;
3) ease of mechanical processing for preparing the parallelepipeds samples.

For each material, two different samples were machined (both taken from the same material block): 1) one to be inserted in the WR430 and 2) the other in the WR90 waveguides. The experimental setup for the WR430 measurement system is shown in Fig. (1(b).

Measurements in both frequency bands were performed using the Agilent PNA E8363C network analyzer. In all the measurements, the TRL error correction procedure was applied, thus compensating for possible nonidealities (such as spurious reflections and wall losses, electrical delay introduced by the sample holder). Throughout the experimental session,
the environmental temperature, monitored through a thermistor sensor, was within the \((26.5 \pm 1.0) ^\circ C\) range.

Table I summarizes the measurement results for the different material samples. In particular, for each tested material, the average value measured for the real part of the permittivity, throughout the entire frequency bandwidth, and the corresponding percentage differences are reported. The results obtained with the proposed WR430 and with the traditional WR90 systems are in good agreement; in fact, the percentage differences between the average permittivities measured through the two systems are below 1%.

V. EXPERIMENTAL RESULTS ON GRANULAR INERT MATERIALS

A. Results on Basaltic and Calcareous Materials

To test the suitability of the proposed WR430 system for dielectric spectroscopy investigations on asphalt materials, measurements were performed on two materials that are typically used in the asphalt industry: 1) calcareous and 2) basaltic aggregates. Both these materials were screened so as to obtain stones with a diameter ranging from 2 to 4 mm.

The total porosity \((\phi)\), defined as the total pore space per unit volume of granular material (including both air contained inside the single grains and air filling the empty spaces between grains) [23], was estimated as follows. A volume of dry material \((V_{\text{dry}})\) equal to the one required to fill the sample holder was placed in a container. Some water was placed in another container and weighed with an electronic balance \((W_b)\). The water was poured inside the material sample, until the material sample was filled with water. The remaining water was then weighed \((W_f)\). The volume of the added water \((V_{\text{w,add}})\) was evaluated as \((W_b - W_f)/\rho_{\text{water}}\), where \(\rho_{\text{water}}\) is the density of water. Finally, the porosity was estimated as \(\phi = V_{\text{w,add}}/V_{\text{dry}}\) [24]. In this way, the \(\phi\) values of the two materials were estimated to be \(\phi_{\text{calc}} = 41.8\%\) for the calcareous material and \(\phi_{\text{bas}} = 49.3\%\) for the basaltic material. The same samples used for estimating porosity were subsequently employed for permittivity measurements. For each material, the dielectric spectroscopy measurements were carried out as follows. Starting from the oven-dried material, the sample was moistened at progressively higher values of volumetric water content \((\theta_{\text{ref}})\)

\[
\theta_{\text{ref}} = \left( \frac{V_{\text{w}}}{V_{\text{dry}}} \right) \times 100 \quad (1)
\]

where \(V_{\text{w}}\) is the volume of the added water.

Table I

<table>
<thead>
<tr>
<th>Material</th>
<th>(\varepsilon_{\text{m, WR430}})</th>
<th>(\varepsilon_{\text{m, WR90}})</th>
<th>(% \Delta \varepsilon_{\text{m}})</th>
</tr>
</thead>
<tbody>
<tr>
<td>LD-PVC</td>
<td>1.619</td>
<td>1.608</td>
<td>0.7%</td>
</tr>
<tr>
<td>PTFE</td>
<td>2.056</td>
<td>2.038</td>
<td>0.9%</td>
</tr>
<tr>
<td>PMMA</td>
<td>2.592</td>
<td>2.591</td>
<td>0.04%</td>
</tr>
<tr>
<td>PC</td>
<td>2.822</td>
<td>2.809</td>
<td>0.5%</td>
</tr>
</tbody>
</table>

For each moistening step, the sample was inserted in the sample holder and shaken so as to ensure that its volume was constant, and the corresponding real part \((\varepsilon'_{\text{m}})\) and imaginary part \((\varepsilon''_{\text{m}})\) of the relative dielectric permittivity were measured with the WR430 system. The actual water content was estimated by weighing the sample immediately after its removal from the sample holder. With regard to the calcareous aggregate, starting from the oven-dried condition, it was moistened at \(\theta_{\text{ref}} = 0.8\%, 1.5\%, 2.1\%, 2.8\%, 4.3\%,\) and, finally, 5.1% (this final value corresponding to near saturation). Fig. 2(a) and (b) shows the \(\varepsilon'_{\text{m}}\) and \(\varepsilon''_{\text{m}}\) results in the considered frequency range, respectively.

Similar experiments were performed on the basaltic material. Staring from the oven-dried material, the sample was moistened at \(\theta_{\text{ref}} = 0.9\%, 1.8\%, 3.0\%, 4.2\%, 6.3\%,\) and, finally, 7.1%. Also in this case, this final value corresponds to near saturation. Fig. 3(a) and (b) shows the \(\varepsilon'_{\text{m}}\) and \(\varepsilon''_{\text{m}}\) results in the considered frequency range, respectively.

For both the considered materials and for each considered \(\theta_{\text{ref}}\) value, the corresponding permittivity values are generally constant in the considered frequency range, thus showing a low dispersivity of the material. Indeed, some fluctuations and abrupt variations are present. As previously discussed, they are attributable to the sample resonances and to the unavoidable
propagation of higher order modes. To better clarify this aspect, the permittivity value (real part) for each sample has been obtained as an average over the entire frequency band, neglecting the regions affected by the resonances. The \( \varepsilon'_m \) values are reported in Tables II and III for the calcareous and basaltic aggregates, respectively. Tables II and III also report the approximate resonance frequencies given by the following equation (by neglecting losses):

\[
f_{\text{res}, k} \approx \frac{1}{\varepsilon'_r} \cdot \left[ \left( \frac{k \cdot c_0}{2 \cdot L} \right)^2 + f_{c0}^2 \right]
\]

where \( c_0 \) is the speed of light in vacuum, \( \varepsilon'_r \) is the real part of the dielectric permittivity of the propagation medium, \( f_{c0} \) is the cutoff frequency of the waveguide in air, \( L \) is the length of the sample, and \( k = 1, 2, 3, \ldots, n \) is the order of the resonance. The computed resonance frequencies agree well with the position of the artifacts on the measured permittivity spectra, appearing as relaxation-like behaviors both in the real and imaginary parts of permittivity. It is worth noting that, in some cases (e.g., dry calcareous aggregate), an apparently impossible increase in permittivity with frequency is visible at the upper edge of the band. However, from the previously computed resonances, it is immediate to verify that such unrealistic behavior is once more the effect of a sample resonance occurring just at the upper edge of the operating frequency band.

Altogether, it can be concluded that, when the system is used for measurements on aggregate materials, the predicted 2% measurement uncertainty is compromised around sample resonance frequencies, but an average permittivity value can still be safely and accurately estimated. Clearly, the system would not be suitable to characterize materials with relaxation phenomena and sample resonances within the explored frequency band. As a last note, some experiments have been performed to assess measurement repeatability, which was always better than 1%. Also system drift, assessed through a measurement in air (empty sample holder) carried out at the end of the measurement session (which could last up to 6 h), showed values of the order of 0.1%.

VI. IDENTIFICATION AND VALIDATION OF THE \( \theta - \varepsilon \)

DIELECTRIC MIXING MODELS

The following step regarded the identification of dielectric mixing models that could suitably describe the \( \theta - \varepsilon \) relationship for the considered materials; in particular, three different models were considered as candidates:

1) a semiempirical model (i.e., the four-component \( \alpha \)-model [25], [26];
2) a probabilistic model (i.e., Ansoult’s model [27], [28]);
3) an empirical model (i.e., Topp’s equation [29]).

These models adopt different approaches to infer the \( \theta - \varepsilon \) relationship. Semiempirical dielectric mixing models [25], [26], [30], [31] are largely used for granular materials. In these models, several parameters are taken into account...
account (such as porosity of the material and distinction of free water from bound water), which are crucial for an accurate physical description of the considered materials, and are sometimes neglected by more general dielectric models.

The probabilistic Ansoult’s model, instead, is based on the random propagation of the pulse in a porous medium that is represented as an array of capacitors. In this approach, the granular material is schematized as a set of three capacitors, each associated with the dielectric constants of the different material constituents (neglecting bound water). A computational algorithm, with a fine-tuning of the so-called degrees of freedom, is used to evaluate the overall equivalent dielectric constant [28].

Finally, empirical approaches simply fit mathematical expressions to measured data: no assumption is made about the state of water in porous materials. In this paper, the well-known Topp’s model was used [29]; hence, a third-order polynomial regression equation was used to represent the $\theta$-ε relationship.

To comparatively assess the robustness and suitability for the specific materials, the following procedure was carried out. First, the $\theta$ values were considered as independent variables of the model, and the corresponding permittivity values ($\varepsilon_{\text{mod}}$) were evaluated from the model $\alpha$ and from Ansoult’s model. These values were then compared with the $\varepsilon_{\text{m}}$ values, directly measured through the WR430 system.

Successively, the dielectric mixing models were inverted. In this way, the measured $\varepsilon_{\text{m}}$ values were considered as independent variables, and the corresponding water content levels $\theta_{\text{mod}}$ were evaluated from the rearranged $\alpha$- and Ansoult’s models. The obtained $\theta_{\text{mod}}$ values were then compared with the known (reference) $\theta$ values. In this case, also Topp’s model was used.

### A. Dielectric Mixing Models

The semiempirical four-component $\alpha$-model is described by the following equation [26], [30], [31]:

$$
\varepsilon_{\text{m}}^\alpha = (\theta - \theta_{\text{bw}}) \varepsilon_{\text{bw}}^\alpha + (1 - \phi) \varepsilon_s^\alpha + \theta_{\text{bw}} \varepsilon_{\text{bw}}^\alpha + (\phi - \theta) \varepsilon_a^\alpha \tag{3}
$$

where $\alpha$ is a parameter that takes into account the geometry and the polarization, $\phi$ is the total porosity, $\varepsilon_{\text{m}}$ is the measured permittivity, $\varepsilon_s$ is the permittivity of the solid material (i.e., without air), $\varepsilon_{\text{bw}}$ is the permittivity of free water (which is equal to 78, at the controlled environmental temperature $27 \pm 1 ^\circ C$), $\varepsilon_a$ is the permittivity of air, $\varepsilon_{\text{bw}}$ is the permittivity of bound water (which was considered equal to 35 [26]), $\theta_{\text{bw}}$ is the volumetric content of (only the) bound water, and, finally, $\theta$ is the total volumetric water content. In particular

$$
\theta_{\text{bw}} = \begin{cases} 
\theta & \text{for } \theta \leq \theta_{\text{bw, max}} \\
\theta_{\text{bw, max}} & \text{for } \theta > \theta_{\text{bw, max}}
\end{cases}
$$

where $\theta_{\text{bw, max}}$ is the saturation value of bound-water content.

The value of $\varepsilon_s$ can be calculated from the permittivity of the dry material ($\varepsilon_{\text{m,dry}}$), by applying (3) and considering $\theta_{\text{bw}} = 0$ and $\theta = 0$:

$$
\varepsilon_s = \left( \frac{\varepsilon_{\text{m,dry}}^\alpha - \phi \cdot \varepsilon_a^\alpha}{1 - \phi} \right)^{1/\alpha}. \tag{4}
$$

In this work, the values of $\alpha$ and $\theta_{\text{bw, max}}$ were evaluated applying a least-squares fitting procedure of the experimental data to (3) and (4). Overall, the following parameter values were obtained: $\varepsilon_s = 5.38$; $\alpha = 0.74$; and $\theta_{\text{bw, max}} = 3\%$ for the basaltic aggregate, and $\varepsilon_s = 5.75$; $\alpha = 0.71$; and $\theta_{\text{bw, max}} = 0.2\%$ for the calcareous aggregate.

It is worth mentioning that the saturation value for bound water could be linked to geometrical characteristics of the grains [26], and thus theoretically computed. On the other hand, also the value assigned to bound water permittivity is somewhat arbitrary, since the different layers of bound water show a progressively increasing permittivity value, and it could be optimized as well. Overall, the obtained values must be considered as a set of best-fitting parameters, suitable to derive a predictive model, rather than representative of the actual geometric structure of the grains.

Starting from these considerations, for each material and for each pre-established volumetric water content level ($\theta$), the corresponding relative dielectric permittivity $\varepsilon_{\text{m,mod}}$ was calculated from (3), obtaining the solid line reported in Fig. 4(a) and (b) for calcareous and basaltic samples, respectively. Successively, Ansoult’s model was applied. In this case, the model requires two inputs, namely, the permittivity of the solid material and the number of degrees of freedom. These parameters were evaluated through a minimization procedure. The obtained number of degrees of freedom was 19 for both materials, while the obtained values of $\varepsilon_s$ were 5.40 and 4.32 for calcareous and basaltic aggregates, respectively. Results of application of the optimized Ansoult’s model are reported in Fig. 4(a) and (b).

Comparing the performances of the two models against the measured values (square markers in Fig. 4), it is immediately evident that both models are suitable for materials, like the tested calcareous aggregate, where bound water plays a minor role (less than 0.2% maximum volumetric content). On the other hand, the four-compartment $\alpha$ model proves superior in cases (like the tested basaltic aggregate) where bound water is a significant fraction (volumetric content up to 3%). In particular, for the calcareous material, the maximum percentage error of the $\alpha$ model evaluated as

$$
\Delta \varepsilon\% = \left( \frac{\varepsilon'_{\text{m,mod}} - \varepsilon'_{\text{m}}}{\varepsilon'_{\text{m}}} \right) \cdot 100
$$

is less than 1%, while it goes up to 1% for Ansoult’s model. For the basaltic sample, instead, the error is less than 1% for the $\alpha$ model and exceeds 10% for Ansoult’s model, which, however, shows performances similar to the $\alpha$ model for volumetric moisture above 3%. It is worth noting that the comparison has been carried out for a range of moisture levels below 10%, while similar comparisons in the literature consider much higher water contents. This is because usually such models are applied to soils, which can be soaked following rain or irrigation. On the other hand, an aggregate used for civil applications, once taken from a stack, can at most be saturated. Therefore, the results point toward the applicability of a four-compartment semiempirical model as a suitable mixing model to describe mineral aggregates used in the asphalt industry in the moisture conditions expected under typical scenarios.
B. Moisture Content Estimation

As aforementioned, to test the robustness of the considered dielectric mixing models, the $\alpha$ model and Ansoult's models were inverted so as to have $\varepsilon_m$ as an independent variable.

In particular, with regard to the $\alpha$-model, the inversion of (3) leads to the following equations:

$$\theta = \begin{cases} 
\frac{\varepsilon_m - \varepsilon_s}{\varepsilon_a} \cdot (1 - \phi) - \frac{\varepsilon_a}{\varepsilon_b} & \text{if } \theta \leq \theta_{bw,\text{max}} \\
\frac{\varepsilon_m - \varepsilon_s}{\varepsilon_a} \cdot (1 - \phi) + \theta_{bw,\text{max}} \cdot (\varepsilon_s - \varepsilon_a) - \phi \cdot \varepsilon_a & \text{if } \theta > \theta_{bw,\text{max}}.
\end{cases}$$

In this case, the $\varepsilon_m$ values were considered as independent variables, thus calculating the corresponding water content values derived from the model ($\theta_{\text{mod}}$).

As for Ansoult's model, to circumvent the fact that its algorithm requires $\theta$ as input (independent variable) and gives $\varepsilon$ as output (dependent variable), without possibility of switching input/output [28], an iterative procedure was carried out. More specifically, different values of $\theta$ were given as input to the algorithm, until the output value of $\varepsilon$ equaled the measured value of the permittivity.

Finally, in addition to the $\alpha$ and Ansoult's models, a totally empirical model (i.e., Topp's model) was used to estimate moisture content. Topp's model is based on a simple third-order polynomial fitting of experimental data. The obtained $\theta_{\text{mod}}$ values for the three models were then compared with the pre-established (true) values of $\theta$.

Tables IV and V summarize the results for the calcareous and basaltic materials, respectively. The results show that, for calcareous aggregate, the three models provide accurate estimates of the water content, while, as expected, Ansoult's model fails for low-moisture contents in the case of the basaltic material. It is worth highlighting that the simple polynomial model (Topp's equation), suitably fitted to experimental data, provides very robust estimations of water content for very different types of materials.

VII. Conclusion

The experimental characterization of the waveguide permittivity measurement system demonstrated that the proposed solution is suitable to perform permittivity measurements in the frequency band around 2.45 GHz, with an uncertainty better than 5%. This performance equals the typical uncertainty of commercial solutions employing open-ended coaxial probes. However, as opposed to the latter open-ended solutions which are by far the most widespread, the proposed system has the great advantage of allowing measurements on granular materials and mixtures employing sample volumes adequate for a thorough macroscopic characterization of the material.

The experimental characterization of the system was completed by performing dielectric permittivity measurements on
inert materials that are typically used in the production of asphalt (calcareous and basaltic aggregates). Finally, to analytically characterize the relationship between water content of the granular materials and the corresponding permittivity values, three different dielectric models (i.e., α model, Ansoult’s model, and Topp’s equation) were comparatively assessed. Experimental tests were performed to identify the most suitable ones in describing the water content and dielectric permittivity relationship for the considered materials. The results not only suggest the need to use a four-compartment model for accurate characterization of the water/aggregate mixture, but also highlight that, as far as moisture estimation from the measured permittivity is the main aim, a third-order polynomial fitting proves an excellent candidate for a simple and ready-to-use model.

The obtained measurement results demonstrate the flexibility and practical usability of the system for possible applications in the road-maintenance related industry.

Compared with standard waveguide systems, the proposed custom solution is characterized by the large sample holder volume, the optimized operating frequency range allowing measurements around the ISM frequency of 2.45 GHz, and the presence of the pressurization windows making the system suitable for granular samples. Overall, the costs for manufacturing the custom-made waveguide components are below €1000, making it a competitive and cost-effective solution.

Finally, it is worth emphasizing that the application of the proposed system is not limited to the aggregate materials used in the asphalt production; on the contrary, it could also be used for dielectric spectroscopy of granular materials in general (with application, for example, in the agri-food industry).

REFERENCES


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AUTHOR QUERIES

AQ:1 = Please provide the expansion for the acronyms “ISM, PNA, CST, and MW.”
AQ:2 = Please confirm whether the edited sentence “The key data needed…” conveys the intended meaning.
AQ:3 = Please confirm whether “coaxial (n-type) to rectangular waveguide” may be modified to read “(n-type) coaxial-to-rectangular waveguide.”
AQ:4 = Please confirm whether the edited sentence “However, it was only…” conveys the intended meaning.
AQ:5 = Please confirm whether the edited sentence “For each material, two…” conveys the intended meaning.
AQ:6 = Please confirm whether “such as spurious reflections and wall losses, electrical delay introduced by the sample holder” may be modified to read “such as spurious reflections and wall losses and electrical delay introduced by the sample holder.”
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Abstract—The design and the experimental characterization of a waveguide system for complex permittivity measurements on both solid and granular materials are presented. The proposed system is intended for the dielectric characterization of asphalt concrete and of its components at frequencies around 2.45 GHz. Therefore, the system provides measurements on granular materials, such as mineral aggregates found in the asphalt concrete. A series of experimental tests on reference materials shows, via comparison with a standard measurement system, errors within 1%. A theoretical analysis of uncertainty contributions confirms a predicted expanded uncertainty lower than 2%. Furthermore, to test the proposed system for dielectric spectroscopy on materials typical of the asphalt industry, measurements were also performed on calcareous and basaltic materials, which are typically used for the production of asphalt. Finally, three different dielectric models (namely, α model, Ansoult’s model, and Topp’s equation) were comparatively assessed to identify the most suitable ones in describing the water content and dielectric permittivity relationship for the considered materials.

Index Terms—Asphalt concrete characterization, permittivity measurements, transmission/reflection methods, uncertainty evaluation, WR430 waveguide.

I. INTRODUCTION

The development of dielectric spectroscopy techniques, with particular emphasis on those operating in the microwave frequency range, has attracted an increasing interest in the last few years [1]. Such techniques permit measurement of the frequency-dependent complex permittivity of a material sample. Knowledge of this quantity can prove useful for quality-control purposes [2], [3] or for understanding the interaction of the material with an electromagnetic wave and the corresponding heating effect [4]. A specific field of application of microwave heating, especially at the 2.45-GHz ISM frequency, is road maintenance [5]. Indeed, by heating the asphalt concrete through the use of microwaves, it is possible to perform on-site repairs of holes or cracks present on the road surface. Still, microwaves might even be employed in the production stage of the asphalt concrete, as a substitute to conventional ovens currently exploited for drying and heating the mineral aggregates, which are subsequently bound together with asphalt. Microwave heating might also help for the recycling process of reclaimed asphalt pavement.

Mineral aggregates used inside the asphalt concrete mixture have different sizes, typically ranging from less than 1 up to 25 mm in diameter. They are obtained from basalt or calcareous rocks. In the final concrete, they are bound together by asphalt (or bitumen), which is usually refined from petroleum. The key data needed for studying the feasibility of a microwave system for asphalt concrete production or recycling are the complex permittivities of the concrete components (asphalt and mineral aggregates) and the concrete itself. Therefore, an accurate system suitable to perform complex permittivity measurements around 2.45 GHz on both compact and granular materials is needed. The most common solutions in this frequency range exploit open-ended coaxial probes [6], whose tip is placed in contact with the material [7]. However, such systems are not suitable to perform measurements on granular materials and have a sensitive area that is definitely too small to allow a correct characterization of asphalt concrete, which is a mixture whose granular components can also be several tens of millimeters in diameter. To overcome such limitation, transmission/reflection solutions can be adopted, where the material is placed inside a suitable section of transmission line [8]. However, currently, implemented systems are based on a coaxial structure [9], [10] and operate in a frequency region well below the ISM band at 2.45 GHz. On the other hand, the most widespread waveguide measurement systems are based on the WR90 standard waveguide, allowing measurements in the 8–12-GHz band (X-band). In this paper, the design and the metrological characterization of a waveguide system, suitable to perform complex permittivity measurements on material samples of...
adequate size, are presented. The system is developed with the aim of providing an accurate and affordable solution for measurement on granular and compact materials in the frequency range around 2.45 GHz.

To test the performance of the proposed system, measurements were also carried out on typical materials used for asphalt, moistened at different water content levels. Finally, different dielectric mixing models were investigated and their suitability for the considered materials was verified.

II. DESIGN OF THE WAVEGUIDE SYSTEM

As reported in [11], the design goal of achieving a permittivity measurement system suitable to characterize adequately sized material samples (possibly granular), suggested the use of a transmission/reflection system based on rectangular waveguides.

Among the different standardized waveguides, the WR430 waveguide, spanning the 1.7–2.6-GHz region, was used. The choice of this waveguide over other standard waveguides with partially overlapping operating frequency range (i.e., the WR340) was due to its larger cross-sectional dimensions (approximately 109 mm × 55 mm), thus accommodating samples with sufficient volume to accurately characterize the asphalt concrete mixture and components.

The whole system was designed through electromagnetic full-wave simulations, performed employing CST Microwave Studio software. A sketch of the designed system is shown in Fig. 1(a). The system is composed of a couple of coaxial (n-type) to rectangular waveguide (WR430) transitions; two standard WR430 waveguide sections (long enough to ensure that higher order modes are sufficiently damped); and a sample holder, 10 cm in length.

Typically, waveguide systems are employed for measurements on solid and compact materials, which can be cut in a parallelepiped shape and inserted in the sample holder. However, one of the goals of the proposed measurement system is the characterization of granular materials (such as the mineral aggregates). Therefore, to allow the insertion of granular samples in the sample holder, two pressurization windows were added, directly connected to the sample holder ends, which are filled by epoxy and confine the material inside the holder. The windows are designed so as to bear up to 1 kg of material. Due to the presence of the epoxy dielectric, a shunt capacitive load is added to the waveguide, thus creating an impedance-mismatch effect. Even though this can be compensated for through standard vector error correction procedures, the windows were equipped with a printed inductive/copper iris. Such an iris, typically realized through two thin rectangular metal patches protruding from the narrow walls of the waveguide (so as to reduce its effective width), is designed to provide a shunt inductance able to cancel, through resonance, the capacitive mismatch at the target frequency of 2.45 GHz [12]. To slightly enlarge the matching bandwidth, the iris was designed with an elliptically shaped (rather than straight) contour, whose eccentricity was optimized through specific electromagnetic simulations. The whole structure is made of brass, with copper-plated inner walls.

To make the system practically usable for performing reflection/transmission measurements with a vector network analyzer, it is necessary to apply the vector error correction procedure at the sample holder ports [13]. Such procedure permits compensation for most of the systematic errors and requires a specific calibration kit. The most simple calibration standards to be employed for waveguide systems are the short circuit and waveguide section standards to be employed for waveguide systems are the short circuit (i.e., a metallic end plate) and a waveguide section of known length (typically equal to one-fourth of the wavelength computed in the middle of the operating frequency band). This set of standards accommodates the thru-reflect-line (TRL) vector error correction procedure. Unfortunately, there are several waveguide calibration kits available on the market; however, to the best of authors’ knowledge, none of them is suitable for the WR430 waveguide format. Therefore, the short circuit and waveguide section standards were built as part of the whole project. Starting from their geometrical and electrical characterization, a custom model for the developed calibration kit was implemented and added to the network analyzer data base [14].

III. MEASUREMENT MODEL AND UNCERTAINTY

In transmission/reflection measurements, the sample is placed inside the waveguide and an electromagnetic wave is launched at the waveguide port. By measuring the scattering parameters at the waveguide ports, it is possible to retrieve the complex permittivity of the sample through an appropriate measurement model.
Historically, the first works that proposed permittivity measurements with the transmission/reflection technique date back to the mid-20th century [15]–[17]. However, it was only in the early 1970s that a new model was developed in [18] and further optimized in [19]: such model, known as Nicolson–Ross–Weir (NRW), accurately operates only at frequencies far from the sample resonances, which occur whenever the sample length becomes an integer multiple of half wavelength computed inside the sample. An alternative model for evaluating the complex permittivity of the sample, starting from the measured scattering parameters, has been developed by the National Institute of Standards and Technology (NIST) [8], [20]: such a model employs a mathematical formulation that largely limits errors related to sample resonances.

In this paper, measurements were performed using an Agilent E8363C vector network analyzer, equipped with Agilent 85071E permittivity measurement software [21]. In particular, this software provides different measurement models, including the aforementioned NRW and NIST models. The measurement results reported herein have been obtained employing the NIST model, which has been proved to be the most accurate for nonmagnetic materials, like the ones of interest for this paper.

A. Uncertainty Contributions

A theoretical evaluation of permittivity measurement uncertainty for the NIST model can be found in [8] and [20]. Applying such evaluation to the proposed system, the following main uncertainty contributions can be identified: 1) uncertainty in scattering parameters; 2) air gaps between sample and holder; 3) dimensional uncertainty in the sample holder; 4) uncertainty in sample length; and 5) uncertainty in the exact sample positioning inside the holder.

In measurements on solid and compact materials, many of these contributions were made negligible thanks to the chosen mechanical tolerances in the system realization and to the accurate milling of the material samples. Therefore, the main remaining contributions are likely those related to scattering parameters and to the sample length. In particular, the uncertainty in sample length is mainly the result of the imperfect planarity of the sample surface, especially for pliable materials.

The exploiting results reported in [8] for the sensitivity coefficients, using uncertainty figures taken from the network analyzer datasheets for scattering parameter measurements, and assuming a worst case uncertainty of 0.05 mm in sample length (on the basis of the measured nonplanarity), the combined uncertainty on the real part of the measured permittivity is about 2% (average value over the entire frequency band). This result is in optimum agreement with typical uncertainty stated in the 85071E software specifications [21].

Clearly, for measurements on granular materials, some of the aforementioned uncertainty contributions may become more prominent. For example, the sample length is harder to be accurately evaluated. In addition, inhomogeneities in the sample may lead to the propagation of higher order modes. Higher order mode propagation, in particular, is responsible for a deterioration in the accuracy of scattering parameter measurements, which become affected by spurious oscillations. These artifacts on the scattering parameters can lead to an increased uncertainty in estimated permittivity, especially around sample resonances, where the sensitivity coefficients for the NIST model tend to become rather large. However, as long as an average value of the permittivity is of interest, because no significant relaxation processes are expected in the measurement bandwidth, artifacts around sample resonances are not of great concern and should not decrease measurement accuracy in a significant way.

IV. EXPERIMENTAL CHARACTERIZATION OF THE PROPOSED SYSTEM

To characterize the presented WR430 custom system, measurements were performed on a set of different solid materials. Unfortunately, while for reference liquid materials, very accurate databases of permittivity are available [22], it is more complex to obtain reliable reference solid materials. Therefore, we used low-permittivity solid materials that are known to exhibit an extremely flat permittivity over the entire microwave frequency band. In this way, it was possible to validate the system through a comparison with measurements performed on the same materials employing a standard WR90 waveguide system operating between 8.2 and 12.4 GHz, and used as a reference. Indeed, the WR90 waveguide system is entirely based on commercial components (with the exception of the sample holder, realized with a milling machine with 0.005 mm repeatability), and it is an excellent candidate to be used as a reference for comparing the WR430 custom system measurements, thanks to its stated typical worst case uncertainty equal to 2% [21].

For the characterization of the system, the following materials were chosen: 1) low-density polyvinyl chloride; 2) polytetrafluoroethylene; 3) polymethyl methacrylate; and 4) polycarbonate. These test materials were chosen to satisfy the following criteria:

1) flat permittivity spectrum so as to allow a direct comparison between the results obtained with the WR430 and the WR90 systems;
2) low permittivity (real part less than 8), in order to better represent permittivities of asphalt concrete main components;
3) ease of mechanical processing for preparing the parallelepiped samples.

For each material, two different samples were machined (both taken from the same material block): 1) one to be inserted in the WR430 and 2) the other in the WR90 waveguides. The experimental setup for the WR430 measurement system is shown in Fig. 1(b).

Measurements in both frequency bands were performed using the Agilent PNA E8363C network analyzer. In all the measurements, the TRL error correction procedure was applied, thus compensating for possible nonidealities (such as spurious reflections and wall losses, electrical delay introduced by the sample holder). Throughout the experimental session,
the environmental temperature, monitored through a thermistor sensor, was within the \((26.5 \pm 1.0) \, ^\circ C\) range.

Table I summarizes the measurement results for the different material samples. In particular, for each tested material, the average value measured for the real part of the permittivity, throughout the entire frequency bandwidth, and the corresponding percentage differences are reported. The results obtained with the proposed WR430 and with the traditional WR90 systems are in good agreement; in fact, the percentage differences between the average permittivities measured through the two systems are below 1%.

V. EXPERIMENTAL RESULTS ON GRANULAR INERT MATERIALS

A. Results on Basaltic and Calcareous Materials

To test the suitability of the proposed WR430 system for dielectric spectroscopy investigations on asphalt materials, measurements were performed on two materials that are typically used in the asphalt industry: 1) calcareous and 2) basaltic aggregates. Both these materials were screened so as to obtain stones with a diameter ranging from 2 to 4 mm.

The total porosity \((\phi)\), defined as the total pore space per unit volume of granular material (including both air contained inside the single grains and air filling the empty spaces between grains) [23], was estimated as follows. A volume of dry material \((V_{\text{dry}})\) equal to the one required to fill the sample holder was placed in a container. Some water was placed in another container and weighed with an electronic balance \((W_b)\). The water was poured inside the material sample, until the material sample was filled with water. The remaining water was then weighed \((W_f)\). The volume of the added water \((V_{\text{a,w}})\) was evaluated as \((W_b - W_f)/\rho_{\text{water}}\), where \(\rho_{\text{water}}\) is the density of water. Finally, the porosity was estimated as \(\phi = V_{\text{a,w}}/V_{\text{dry}}\) [24]. In this way, the \(\phi\) values of the two materials were estimated to be \(\phi_{\text{calc}} = 41.8\%\) for the calcareous material and \(\phi_{\text{bas}} = 49.3\%\) for the basaltic material. The same samples used for estimating porosity were subsequently employed for permittivity measurements. For each material, the dielectric spectroscopy measurements were carried out as follows. Starting from the oven-dried material, the sample was moistened at progressively higher values of volumetric water content \((\theta_{\text{ref}})\):

\[
\theta_{\text{ref}} = \left( \frac{V_{\text{a,w}}}{V_{\text{dry}}} \right) \times 100
\]  

where \(V_{\text{a,w}}\) is the volume of the added water.

<table>
<thead>
<tr>
<th>Material</th>
<th>(\varepsilon''_{m,W430})</th>
<th>(\varepsilon''_{m,W90})</th>
<th>%(\Delta\varepsilon''_{m})</th>
</tr>
</thead>
<tbody>
<tr>
<td>LD-PVC</td>
<td>1.619</td>
<td>1.608</td>
<td>0.7%</td>
</tr>
<tr>
<td>PTFE</td>
<td>2.056</td>
<td>2.038</td>
<td>0.9%</td>
</tr>
<tr>
<td>PMMA</td>
<td>2.592</td>
<td>2.591</td>
<td>0.04%</td>
</tr>
<tr>
<td>PC</td>
<td>2.822</td>
<td>2.809</td>
<td>0.5%</td>
</tr>
</tbody>
</table>

Fig. 2. Measurement results on the calcareous aggregate for different water content levels. (a) Real and (b) imaginary parts of the relative dielectric permittivity.
propagation of higher order modes. To better clarify this aspect, the permittivity value (real part) for each sample has been obtained as an average over the entire frequency band, neglecting the regions affected by the resonances. The ε′ m values are reported in Tables II and III for the calcareous and basaltic aggregates, respectively. Tables II and III also report the approximate resonance frequencies given by the following equation (by neglecting losses):

\[ f_{res,k} \approx \sqrt{\frac{1}{\varepsilon'_r} \left( \frac{k \cdot c_0}{2 \cdot L} \right)^2 + f_{c0}^2} \]  

(2)

where \( c_0 \) is the speed of light in vacuum, \( \varepsilon'_r \) is the real part of the dielectric permittivity of the propagation medium, \( f_{c0} \) is the cutoff frequency of the waveguide in air, \( L \) is the length of the sample, and \( k = 1, 2, 3, \ldots, n \) is the order of the resonance. The computed resonance frequencies agree well with the position of the artifacts on the measured permittivity spectra, appearing as relaxation-like behaviors both in the real and imaginary parts of permittivity. It is worth noting that, in some cases (e.g., dry calcareous aggregate), an apparently impossible increase in permittivity with frequency is visible at the upper edge of the band. However, from the previously computed resonances, it is immediate to verify that such unrealistic behavior is once more the effect of a sample resonance occurring just at the upper edge of the operating frequency band.

Altogether, it can be concluded that, when the system is used for measurements on aggregate materials, the predicted 2% measurement uncertainty is compromised around sample resonance frequencies, but an average permittivity value can still be safely and accurately estimated. Clearly, the system would not be suitable to characterize materials with relaxation phenomena and sample resonances within the explored frequency band. As a last note, some experiments have been performed to assess measurement repeatability, which was always better than 1%. Also system drift, assessed through a measurement in air (empty sample holder) carried out at the end of the measurement session (which could last up to 6 h), showed values of the order of 0.1%.

VI. IDENTIFICATION AND VALIDATION OF THE θ – ε DIELECTRIC MIXING MODELS

The following step regarded the identification of dielectric mixing models that could suitably describe the \( \theta – \varepsilon \) relationship for the considered materials; in particular, three different models were considered as candidates:

1) a semiempirical model (i.e., the four-component \( \alpha \)-model [25], [26]);
2) a probabilistic model (i.e., Ansoult's model [27], [28]);
3) an empirical model (i.e., Topp’s equation [29]).

These models adopt different approaches to infer the \( \theta – \varepsilon \) relationship. Semiempirical dielectric mixing models [25], [26], [30], [31] are largely used for granular materials. In these models, several parameters are taken into account.
account (such as porosity of the material and distinction of free water from bound water), which are crucial for an accurate physical description of the considered materials, and are sometimes neglected by more general dielectric models.

The probabilistic Ansoult’s model, instead, is based on the random propagation of the pulse in a porous medium that is represented as an array of capacitors. In this approach, the granular material is schematized as a set of three capacitors, each associated with the dielectric constants of the different material constituents (neglecting bound water). A computational algorithm, with a fine-tuning of the so-called degrees of freedom, is used to evaluate the overall equivalent dielectric constant [28].

Finally, empirical approaches simply fit mathematical expressions to measured data: no assumption is made about the state of water in porous materials. In this paper, the well-known Topp’s model was used [29]; hence, a third-order polynomial regression equation was used to represent the \( \theta - \varepsilon \) relationship.

To comparatively assess the robustness and suitability for the specific materials, the following procedure was carried out. First, the \( \theta \) values were considered as independent variables of the model, and the corresponding permittivity values \( (\varepsilon_{m,\text{mod}}) \) were evaluated from the model \( \alpha \) and from Ansoult’s model. These values were then compared with the \( \varepsilon_m \) values, directly measured through the WR430 system. Successively, the dielectric mixing models were inverted. In this way, the measured \( \varepsilon_m \) values were considered as independent variables, and the corresponding water content levels \( \theta_{\text{mod}} \) were evaluated from the rearranged \( \alpha \)- and Ansoult’s models. The obtained \( \theta_{\text{mod}} \) values were then compared with the known (reference) \( \theta \) values. In this case, also Topp’s model was used.

### A. Dielectric Mixing Models

The semiempirical four-component \( \alpha \)-model is described by the following equation [26], [30], [31]:

\[
\varepsilon_m^{\alpha} = (\theta - \theta_{bw})\varepsilon_{bw}^{\alpha} + (1 - \phi)\varepsilon_a + \theta_{bw}\varepsilon_{bw} + (\phi - \theta)\varepsilon_a^{\alpha} \tag{3}
\]

where \( \alpha \) is a parameter that takes into account the geometry and the polarization, \( \phi \) is the total porosity, \( \varepsilon_m^{\alpha} \) is the measured permittivity, \( \varepsilon_a \) is the permittivity of the solid material (i.e., without air), \( \varepsilon_{bw} \) is the permittivity of free water (which is equal to 78, at the controlled environmental temperature \( 27 \pm 1 \) \(^\circ\) C), \( \varepsilon_a \) is the permittivity of air, \( \varepsilon_{bw} \) is the permittivity of bound water (which was considered equal to 35 [26]), \( \theta_{bw} \) is the volumetric content of (only the) bound water, and, finally, \( \theta \) is the total volumetric water content. In particular

\[
\theta_{bw} = \begin{cases} 
\theta & \text{for } \theta \leq \theta_{bw,\text{max}} \\
\theta_{bw,\text{max}} & \text{for } \theta > \theta_{bw,\text{max}} 
\end{cases}
\]

where \( \theta_{bw,\text{max}} \) is the saturation value of bound-water content.

The value of \( \varepsilon_s \) can be calculated from the permittivity of the dry material \( (\varepsilon_{m,\text{dry}}) \), by applying (3) and considering \( \theta_{bw} = 0 \) and \( \theta = 0 \):

\[
\varepsilon_s = \left( \frac{\varepsilon_{m,\text{dry}}}{\varepsilon_a} - \frac{\phi \cdot \varepsilon_a^{\alpha}}{1 - \phi} \right)^{1/\alpha} \tag{4}
\]

In this work, the values of \( \alpha \) and \( \theta_{bw,\text{max}} \) were evaluated applying a least-squares fitting procedure of the experimental data to (3) and (4). Overall, the following parameter values were obtained: \( \varepsilon_s = 5.38, \alpha = 0.74 \); and \( \theta_{bw,\text{max}} = 3\% \) for the basaltic aggregate, and \( \varepsilon_s = 5.75, \alpha = 0.71 \); and \( \theta_{bw,\text{max}} = 0.2\% \) for the calcareous aggregate.

It is worth mentioning that the saturation value for bound water could be linked to geometrical characteristics of the grains [26], and thus theoretically computed. On the other hand, also the value assigned to bound water permittivity is somewhat arbitrary, since the different layers of bound water show a progressively increasing permittivity value, and it could be optimized as well. Overall, the obtained values must be considered as a set of best-fitting parameters, suitable to derive a predictive model, rather than representative of the actual geometric structure of the grains.

Starting from these considerations, for each material and for each pre-established volumetric water content level \( (\theta) \), the corresponding relative dielectric permittivity \( \varepsilon'_m,\text{mod} \) was calculated from (3), obtaining the solid line reported in Fig. 4(a) and (b) for calcareous and basaltic samples, respectively. Successively, Ansoult’s model was applied. In this case, the model requires two inputs, namely, the permittivity of the solid material and the number of degrees of freedom. These parameters were evaluated through a minimization procedure. The obtained number of degrees of freedom was 19 for both materials, while the obtained values of \( \varepsilon_s \) were 5.40 and 4.32 for calcareous and basaltic aggregates, respectively. Results of application of the optimized Ansoult’s model are reported in Fig. 4(a) and (b).

Comparing the performances of the two models against the measured values (square markers in Fig. 4), it is immediately evident that both models are suitable for materials, like the tested calcareous aggregate, where bound water plays a minor role (less than 0.2% maximum volumetric content). On the other hand, the four-compartment \( \alpha \)-model proves superior in cases (like the tested basaltic aggregate) where bound water is a significant fraction (volumetric content up to 3%). In particular, for the calcareous material, the maximum percentage error of the \( \alpha \) model evaluated as

\[
\Delta \varepsilon\% = \left( \frac{|\varepsilon'_m,\text{mod} - \varepsilon'_m|^* \cdot 100}{\varepsilon'_m} \right)
\]

is less than 1%, while it goes up to 1% for Ansoult’s model. For the basaltic sample, instead, the error is less than 1% for the \( \alpha \) model and exceeds 10% for Ansoult’s model, which, however, shows performances similar to the \( \alpha \) model for volumetric moisture above 3%. It is worth noting that the comparison has been carried out for a range of moisture levels below 10%, while similar comparisons in the literature consider much higher water contents. This is because usually such models are applied to soils, which can be soaked following rain or irrigation. On the other hand, an aggregate used for civil applications, once taken from a stack, can at most be saturated. Therefore, the results point toward the applicability of a four-compartment semiempirical model as a suitable mixing model to describe mineral aggregates used in the asphalt industry in the moisture conditions expected under typical scenarios.
Fig. 4. Comparison of the results obtained applying different dielectric models. (a) Calcareous and (b) basaltic aggregates.

**B. Moisture Content Estimation**

As aforementioned, to test the robustness of the considered dielectric mixing models, the $\alpha$ model and Ansoult's models were inverted so as to have $\varepsilon_m$ as an independent variable.

In particular, with regard to the $\alpha$-model, the inversion of (3) leads to the following equations:

$$\theta = \begin{cases} \frac{\varepsilon^a_m - \varepsilon^a_s \cdot (1 - \phi) - \phi \cdot \varepsilon^a_a}{\varepsilon^a_{bw} - \varepsilon^a_a} & \text{if } \theta \leq \theta_{bw,max} \\ \frac{\varepsilon^a_m - \varepsilon^a_s \cdot (1 - \phi) + \theta_{bw,max} \cdot (\varepsilon^a_{fw} - \varepsilon^a_{bw}) - \phi \cdot \varepsilon^a_a}{\varepsilon^a_{fw} - \varepsilon^a_a} & \text{if } \theta > \theta_{bw,max} \end{cases}$$

In this case, the $\varepsilon_m$ values were considered as independent variables, thus calculating the corresponding water content values derived from the model ($\theta_{mod}$).

As for Ansoult's model, to circumvent the fact that its algorithm requires $\theta$ as input (independent variable) and gives $\varepsilon$ as output (dependent variable), without possibility of switching input/output [28], an iterative procedure was carried out. More specifically, different values of $\theta$ were given as input to the algorithm, until the output value of $\varepsilon$ equaled the measured value of the permittivity.

Finally, in addition to the $\alpha$ and Ansoult's models, a totally empirical model (i.e., Topp's model) was used to estimate moisture content. Topp's model is based on a simple third-order polynomial fitting of experimental data. The obtained $\theta_{mod}$ values for the three models were then compared with the pre-established (true) values of $\theta$.

Tables IV and V summarize the results for the calcareous and basaltic materials, respectively. The results show that, for calcareous aggregate, the three models provide accurate estimates of the water content, while, as expected, Ansoult's model fails for low-moisture contents in the case of the basaltic material. It is worth highlighting that the simple polynomial model (Topp's equation), suitably fitted to experimental data, provides very robust estimations of water content for very different types of materials.

**VII. Conclusion**

The experimental characterization of the waveguide permittivity measurement system demonstrated that the proposed solution is suitable to perform permittivity measurements in the frequency band around 2.45 GHz, with an uncertainty better than 5%. This performance equals the typical uncertainty of commercial solutions employing open-ended coaxial probes. However, as opposed to the latter open-ended solutions which are by far the most widespread, the proposed system has the great advantage of allowing measurements on granular materials and mixtures employing sample volumes adequate for a thorough macroscopic characterization of the material.

The experimental characterization of the system was completed by performing dielectric permittivity measurements on
inert materials that are typically used in the production of asphalt (calcareous and basaltic aggregates). Finally, to analytically characterize the relationship between water content of the granular materials and the corresponding permittivity values, three different dielectric models (i.e., α model, Ansoult’s model, and Topp’s equation) were comparatively assessed. Experimental tests were performed to identify the most suitable ones in describing the water content and dielectric permittivity relationship for the considered materials. The results not only suggest the need to use a four-compartment model for accurate characterization of the water/aggregate mixture, but also highlight that, as far as moisture estimation from the measured permittivity is the main aim, a third-order polynomial fitting proves an excellent candidate for a simple and ready-to-use model.

The obtained measurement results demonstrate the flexibility and practical usability of the system for possible applications in the road-maintenance related industry.

Compared with standard waveguide systems, the proposed custom solution is characterized by the large sample holder volume, the optimized operating frequency range allowing measurements around the ISM frequency of 2.45 GHz, and the presence of the pressurization windows making the system suitable for granular samples. Overall, the costs for manufacturing the custom-made waveguide components are below €1000, making it a competitive and cost-effective solution.

Finally, it is worth emphasizing that the application of the proposed system is not limited to the aggregate materials used in the asphalt production; on the contrary, it could also be used for dielectric spectroscopy of granular materials in general (with application, for example, in the agri-food industry).

REFERENCES


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