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# Characterization of a non-destructive microwave technique for the detection and quantification of water in thermosets and thermoplastics

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ABSTRACT

In this paper, the detection and quantification of the moisture content in epoxy and polyamide-6 using a non-destructive and portable device based on a microwave resonator technique is reported. Parameters related to the real ($\varepsilon'$) and imaginary ($\varepsilon''$) part of the complex permittivity were used to quantify the moisture content in these polymers. The maximal penetration and analyzing depth of the microwaves through polyamide-6 (both at least of 7.35 mm) and epoxy (24 mm and 12 mm, respectively) were measured performing samples with different thicknesses. Moreover, measurements were performed at different distances from the sample surface being possible to perform contactless measurements.



## 1. Introduction

The moisture content in materials is actually considered a fundamental property of a component and often is a requirement in order to fulfill quality standards. The presence of water in polymer materials like polyamide or epoxy can be a problem in different applications (*e.g.* adhesive bonding or extrusion processes) from a technical and economical point of view [1].

Non-destructive techniques for moisture detection were at first based on the relation between the electrical conductivity in a material and its moisture content. These methods were soon supplemented by methods using electromagnetic radiation, for example, microwaves. The first water detectors based on microwaves were applied in agriculture and food industry [2–4]. Later on, microwave devices were also adapted to and applied in the tobacco and rice sectors [5–7].

Also several other techniques have already been used to analyze the moisture content in polymers, like nuclear magnetic resonance (NMR) [8] or infrared spectroscopy (IR) [9]. IR and NMR are not only accurate methods for quantifying water, but also provide information at molecular scale about interactions between polymer chains and water [9]. However, their analyzing depths, defined as the depth at which the power density has decreased to 37% of its initial value at the surface, are usually much lower than for the used microwave device, which can also measure moisture content inside the material [10]. Moreover, the water characteristic frequencies in the IR spectrum are very often overlapped by other bands of the polymer chain or surface contaminants. Gravimetric methods are very useful as reference techniques but they are considered as destructive tests. However, the gravimetric method is always used to correlate moisture content in materials with different parameters such as physical or chemical material properties. The quantification of the moisture content in a material through its physical or chemical properties is understood as an indirect measurement method.

Moisture measurements using dielectric methods in the microwave range are indirect measuring methods. The complex permittivity of a tested material can be measured by different microwave techniques. It is not only influenced by the moisture content *MC*, but also by density variations $\Delta\rho$ and temperature $T$, among others. In microwave transmission measurements, the attenuation $A$ and phase shift $\varphi$ are used as parameters which are closely related to the relative permittivity. In resonator-based techniques, the frequency shift $\Delta f_R$ and the change of bandwidth $\Delta B$ are widely used, both also depend on the relative permittivity. Methods based

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on one or two parameters using transmission or resonant properties have been developed [1].

The application of microwave techniques in agriculture, food, tobacco, and rice industries is due to the numerous efforts that have been devoted to [2–7]; however, in materials science technological applications for detecting and quantifying water are sparse [1].

In this paper, we present the results for moisture content detection and quantification in polymers using a two-parameter method and resonator technique. Moreover, the device was characterized, determining the detection limits of moisture, the penetration and analyzing depth of the microwaves through the materials, and the influence of the distance between the sensor and the surface of the polymers on the signal.

## 2. Experimental part

In the following chapter, the sample preparation, sample conditioning and the measuring methods for the different experiments are described for an epoxy resin and polyamide-6.

### 2.1. Sample preparation

Epoxy samples were prepared using diglycidyl ether of Bisphenol A (DGEBA, Araldit GY250 from Hartmann) as resin and isophorone diamine (IPDA) as hardener with a molar ratio of 1:1. The components were mixed for 90 s at 3000 rpm using a SpeedMixer™ DAC 150 FVZ and filled in square silicone molds ($10 \times 10$ cm$^2$) and cured (thicknesses from 0.96 mm to 32.99 mm). The curing cycle consisted of three steps: (1) heating from RT to 140 °C for 57 min; (2) at 140 °C for 1 h; and (3) cooling down to RT [11].

Thin polyamide-6 samples (nylon-6, thickness 0.35 mm) were purchased from Goodfellow (AM301350) [12]. They were cut in smaller samples ($7 \times 7$ cm$^2$).

### 2.2. Conditioning

For the preparation of the epoxy samples with specific moisture content, the cured samples were first dried in a sealed box at 70 °C using silica gel until the sample mass was constant. The dried samples were then stored at 70 °C at a specific relative humidity (0% rh, 11% rh, 30% rh, 50% rh, 62% rh, 75% rh, and 95% rh, and immersed in water). The different relative humidites were generated using saturated salt solutions (LiCl (11% rh), $MgCl_2 \cdot 6\,H_2O$ (30% rh), NaBr (50% rh), KI (62% rh), NaCl (75% rh), $K_2SO_4$ (95% rh)) [13]. After four weeks, the samples reached constant mass and the gain of mass (water uptake) was gravimetrically determined at room temperature. The moisture content ($MC$) was then calculated as shown in Eq. 1:

$$MC = \frac{m_w}{m_d} \cdot 100 \tag{1}$$

where $m_w$ is the water uptake and $m_d$ is the mass of the dried material.

Thin polyamide-6 film samples with specific $MC$ were prepared accordingly. But in this case, it was important to avoid longer storage at high temperature and at high relative humidity because this leads to a degradation reaction of the polyamide-6 [14]. Therefore all polyamide-6 samples were measured directly after 48 h of saturation. In Table 1, the final moisture contents ($MC$) of the epoxy and polyamide-6 samples are given.

**Table 1**
Moisture content depending on the relative humidity for epoxy and polyamide-6.

| Relative humidity [%] | $MC_{Epoxy}$ [wt%] | $MC_{Polyamide\text{-}6}$ [wt%] |
|---|---|---|
| 0 | 0.00 | 0.00 |
| 11 | 0.13 | 0.56 |
| 30 | 0.54 | 2.04 |
| 50 | 0.92 | 2.74 |
| 62 | 1.11 | 3.73 |
| 75 | 1.43 | 3.91 |
| 95 | 2.11 | 6.03 |
| In water | 2.48 | 8.14 |

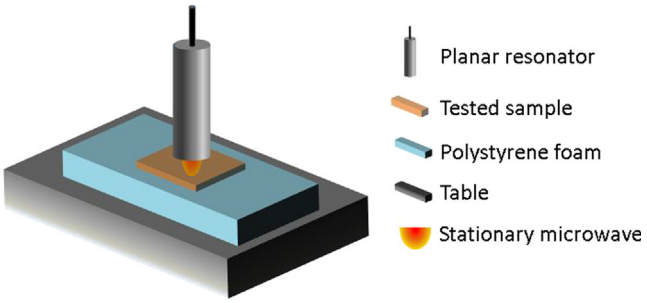


**Fig. 1.** Set-up of the microwave measurement.

### 2.3. Measuring method

The interaction of electromagnetic waves with non-magnetic materials can be described by the complex permittivity $\varepsilon = \varepsilon' + j\varepsilon''$ which is a scalar for isotropic materials. In general, the permittivity of a given material is a function of the frequency $f$, temperature $T$, and density of the dry material $\rho_d$. For a moisture-containing material, $\varepsilon$ will additionally depend on the moisture content for a given frequency $f$ and temperature $T$, as shown by Meyer and Schilz [6]

$$\varepsilon(\rho_d, MC) = \varepsilon'(\rho_d, MC) + j\varepsilon''(\rho_d, MC) \tag{2}$$

The real part $\varepsilon'$of the relative permittivity of free water at 20 GHz is about 80 while most of the usual porous solids exhibit values around 2 and 7. This is why microwave techniques are quite sensitive to moisture.

Based on experimental results, the following empirical equations for $\varepsilon'$ and $\varepsilon''$ have been derived [4]

$$\varepsilon' - 1 = f_1(MC) \cdot g_1(\rho_d);\ \varepsilon'' = f_2(MC) \cdot g_2(\rho_d) \tag{3}$$

where $g_1$ and $g_2$ are functions of density and $f_1$ and $f_2$ are functions of $MC$.

Assuming that the density functions $g_1$ and $g_2$ can be approximated by linear functions or linearly related power series, the following density-independent function can be derived [6]:

$$\frac{\varepsilon' - 1}{\varepsilon''} = \frac{f_1(MC) \cdot g_1(\rho_d)}{f_2(MC) \cdot g_2(\rho_d)} \approx \text{constant} \cdot \frac{f_1(MC)}{f_2(MC)} = K \cdot f(MC) \tag{4}$$

with $K$ as proportionality constant and $f(MC)$ as function depending on the moisture content.

Using microwave resonator techniques, the shift of the resonant frequency ($\Delta f_R$) and the change of the full width at half maximum ($\Delta B$) are measured according to the 'Two-Parameter Method' developed by Meyer and Schilz. Based on the linearity observed experimentally between $\Delta f_R$ and $\Delta B$ by changing the material mass, a density-independent function to measure moisture in solid