

Rapid Communication

Complex permittivities of cyclomaltooligosaccharides (cyclodextrins) over microwave frequencies to 26 GHz

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Abstract

Complex permittivities (ϵ^*) for microwave radiation between 0.5 and 26 GHz have been determined for α -, β -, and γ -cyclodextrins in the solid state at room temperature. For the real component of ϵ^* , maxima occur near 0.6 GHz, and the relation $\beta > \alpha > \gamma$ is evident across the full-frequency spectrum. Dielectric loss is significant only between 5 and 12 GHz for β - and γ -cyclodextrins with maxima near 7.5 GHz. © 2000 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Due to the rapidly increasing utilization of microwave power sources for dielectric heating, the need to understand the relative susceptibilities of materials to microwave irradiation is also growing. Here we report the complex permittivities for cyclomaltohexaose, cyclomaltoheptaose and cyclomaltooctaose

(α -, β -, and γ -cyclodextrins) in the solid state over the microwave frequency range between 0.5 and 26 GHz. Cyclodextrins are naturally occurring toroidal oligosaccharides formed by α -(1 \rightarrow 4) linkage of D-glucopyranose units. α -, β -, and γ -Cyclodextrins are comprised of six, seven, and eight, D-glucose monomers, respectively [1,2]. Cyclodextrins and derivatized cyclodextrins are frequently employed to render lipophilic materials soluble in water and as sorption media for chiral chromatographic separations [3,4].

In solids, the absorption of microwaves occurs through polarizations, which are induced by a variety of mechanisms [5,6]. Hence, it is customary to refer to the complex permittivity of the material,

$$\epsilon^* = \epsilon' - i\epsilon'' \quad (1)$$

where the complex ‘relative’ permittivity (ϵ^*/ϵ_0) is equivalent to the dielectric constant. In Fig. 1, the real and imaginary components of ϵ^* are illustrated in the complex plane. The

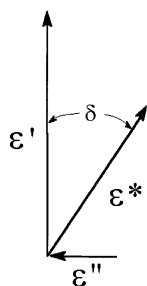


Fig. 1. Permittivity in the complex plane.

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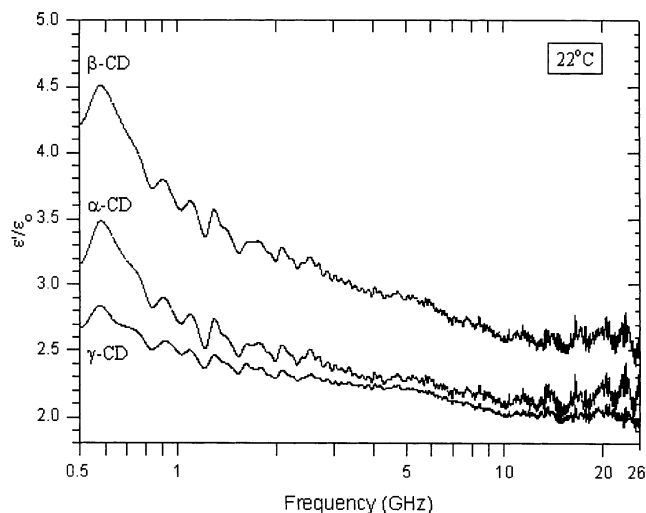


Fig. 2. Real component of cyclodextrin complex relative permittivities.

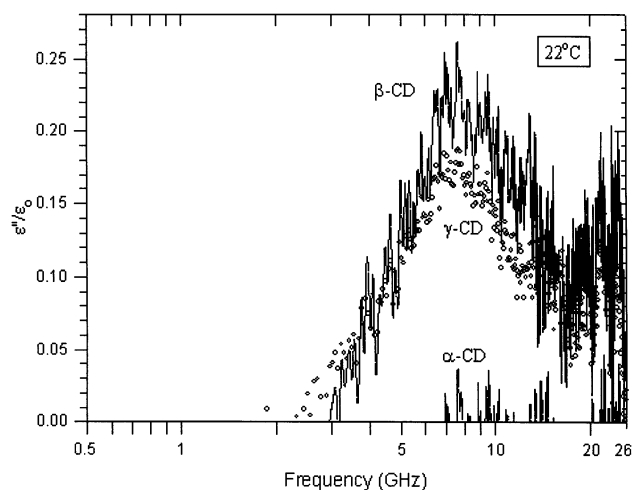


Fig. 3. Imaginary component of cyclodextrin complex permittivities.

loss factor or dissipation factor ($\tan \delta$) is defined as,

$$\tan \delta = \frac{\omega \varepsilon'' E_o^2}{\omega \varepsilon' E_o^2} = \frac{\varepsilon''}{\varepsilon'} \quad (2)$$

where E is electric field strength and ω is angular frequency. The average power per unit volume (P) consumed by loss mechanisms can then be calculated as,

$$P = 1/2 \omega \varepsilon'' E_o^2 \quad (3)$$

2. Results and discussion

The complex permittivity experiments were conducted using a vector network analyzer based test system consisting of an HP8722D Vector Network Analyzer (VNA), HP85070B Dielectric Probe, HP85056A calibration kit, and HP85130F adapter kit [7,8]. While the VNA was capable of operation between 0.05 and 40 GHz, limitations of the dielectric probe and associated transmission elements reduced the practical working frequency range to 0.2–26 GHz. Complex permittivities were calculated from the measured transmission and reflection parameters using HP85071B software. Dissipation factors ≥ 0.05 are required to assure accuracy of the test results.

To confirm the validity of data derived using this system, the complex permittivities of aqueous 1,5-pentanediol solutions were determined from 0.5 to 26 GHz at 22 °C. Polarization relaxation times and static dielectric constants calculated using these data agreed closely with those derived using a circular waveguide/interferometric technique [9].

Cyclodextrins were provided by Cerestar USA, Inc. To minimize interference from adsorbed water, each material was dried at elevated temperature for a minimum of 72 h and stored in a desiccator prior to analysis. Samples were presented to the instrument at ambient temperature as fine powders, thus obviating the need to correct for the effects of intergranular porosity [10,11]. Replicate measurements yielded relative standard deviations $\leq 1\%$ across the full-frequency spectrum. The real and imaginary components of the complex permittivities over the frequency range between 0.5 and 26 GHz are presented in Figs. 2 and 3, respectively. For convenience, the numerical values are given as dimensionless 'relative permittivities' (i.e., $\varepsilon'/\varepsilon_o$, and $\varepsilon''/\varepsilon_o$). The corresponding dissipation factors are summarized in Table 1. Adequacy of drying is evident from the absence of contributions characteristic of water.

The real components of the complex permittivities show the same overall trends across the full-frequency spectrum for the three cyclodextrins, with the relative magnitudes given by the relation: $\beta > \alpha > \gamma$. Maxima occur near

Table 1
Dissipation factors for cyclodextrin powder

<i>f</i> (GHz)	tan δ (ϵ''/ϵ')		
	α -CD	β -CD	γ -CD
0.5	<0.05	<0.05	<0.05
1.0	<0.05	<0.05	<0.05
2.0	<0.05	<0.05	<0.05
2.5	<0.05	<0.05	<0.05
3.0	<0.05	<0.05	<0.05
5.0	<0.05	0.057	0.056
7.5	<0.05	0.083	0.085
10.0	<0.05	0.065	0.066
12.5	<0.05	0.062	0.050
15.0	<0.05	<0.05	<0.05
17.5	<0.05	<0.05	<0.05
20.0	<0.05	<0.05	<0.05
22.0	<0.05	0.068	0.075
24.0	<0.05	<0.05	<0.05
26.0	<0.05	<0.05	<0.05

0.6 GHz. The imaginary component indicates that microwave absorption occurs between 5 and 12 GHz for β - and γ -cyclodextrins, which is strongest between 6 and 10 GHz, and with maxima near 7.5 GHz. No significant dielectric loss is evident for α -cyclodextrin over this frequency range. The overall trend follows: $\beta > \gamma > \alpha$. These data indicate that, while α -cyclodextrin is relatively impervious to microwave absorption, β - and γ -cyclodextrins are susceptible at frequencies above 4 GHz,

particularly within the range between 5 and 12 GHz.

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References

- [1] K.A. Connors, *Chem. Rev.*, 97 (1997) 1325–1357.
- [2] C.J. Easton, S.F. Lincoln, *Modified Cyclodextrins: Scaffolds and Templates for Supramolecular Chemistry*, Imperial College Press, London, 1999.
- [3] F.W. Lichtenthaler, S. Immel, *Justus Liebigs Ann. Chem.*, (1996) 27–37.
- [4] F.W. Lichtenthaler, S. Immel, *J. Inclusion Phenom. Mol. Recogn. Chem.*, 25 (1996) 3–16.
- [5] A. von Hippel, *Dielectrics and Waves*, Wiley, New York, 1954.
- [6] A. von Hippel (Ed.), *Dielectric Materials and Applications*, Technology Press of MIT, Cambridge, 1954.
- [7] J. Lou, A.K. Paravastu, P.E. Laibinis, A.T. Hatton, *J. Phys. Chem. A*, 101 (1997) 9892–9899.
- [8] J. Lou, T.A. Hatton, P.E. Laibinis, *J. Phys. Chem. A*, 101 (1997) 5262–5268.
- [9] F. Wang, R. Pottel, U. Kaatzefind, *J. Phys. Chem. B*, 101 (1997) 922–929.
- [10] D.J. Bergman, *Phys. Lett. C*, 43 (1978) 77–407.
- [11] D. Polder, J.H. Van Santen, *Physica*, 12 (1946) 257–271.