JOURNAL OF SCIENCE
SAKARYA UNIVERSITY

Sakarya University Journal of Science

ISSN 1301-4048 | e-ISSN 2147-835X | Period Bimonthly | Founded: 1997 | Publisher Sakarya University | http://www.saujs.sakarya.edu.tr/en/

Title: Synthesis and Dielectric Properties of Magnesium Silicate Hydrate Deposited With SnO2

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Recieved: 2019-12-16 13:35:20

Accepted: 2020-02-22 17:23:25

Article Type: Research Article
Volume: 24
Issue: 3
Month: June
Year: 2020
Pages: 455-459

How to cite
Yüksel AKINAY; (2020), Synthesis and Dielectric Properties of Magnesium Silicate Hydrate Deposited With SnO2. Sakarya University Journal of Science, 24(3), 455-459, DOI: https://doi.org/10.16984/saufenbilder.659958
Access link
http://www.saujs.sakarya.edu.tr/en/issue/52472/659958

Sakarya University Journal of Science 24(3), 455-459, 2020

Synthesis and Dielectric Properties of Magnesium Silicate Hydrate Deposited With SnO_2

Yüksel AKINAY*[1]

Abstract

In this study, the dielectric properties of SnO_2 coated Magnesium Silicate Hydrate (MSH) pigments were evaluated. The SnO_2 coated MSH pigments were obtained by chemical reduction method with the 3:7 SnO_2/MSH ratio by weight. The structural and dielectric properties of this pigment and MSH were investigated. The surface morphology and phase types were determined by scanning electron microscope (SEM) and x-ray diffraction (XRD). The bond types were characterized by Fourier Transform Infrared Spectrophotometer (FT-IR). The surface of MSH was uniformly coated with SnO_2 as accepted in the SEM images. The existence of XRD peaks for SnO_2 nanoparticles proves the presence of SnO_2 coating. The dielectric properties of prepared pigments were measured via vector network analyzer (VNA) in the frequency range of 8.2–12.4 GHz (X-Band). The dielectric properties of SnO_2 deposited MSH pigments were obtained to be about almost 3-4 times than MSH in the 8-12 GHz frequency range. This study is the first report for the dielectric properties of SnO_2 deposited MSH pigments.

Keywords: Magnesium silicate hydrate, SnO_2, permittivity, pearlescent pigments

1. INTRODUCTION

Pearlescent pigments found naturally and synthetically show outstanding color properties based on optical thin layers. The inorganic pearlescent pigments are obtained by coating a low refractive substrate with high refractive metal oxides. Furthermore, these pigments have superior performance such as thermal and UV stability [1-3]. Inorganic pigments have been widely used in industrial fields such as enamel, ink, plastics, cosmetics and printed products. In recent years, solar reflective and thermal coatings have attracted great attention because they can reduce solar heat in the building and improve indoor thermal conditions, reducing demand for air-conditioning buildings [4,5]. However, there is increasing requests for dielectric properties in the civil and military application such as electromagnetic wave absorber or shielding. Because the high dielectric loss tangent provides

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high microwave absorption performance for the defense and civil industries. [6].

Magnesium silicate hydrate (MSH) is a typical mineral that composed the MgO-SiO_2-H_2O system and forms in a short time at room temperature when $MgO/Mg(OH)_2$ containing silicate layer contact with water [7]. The chemical reactions M-S-H gel at room temperature is shown in Eq. (1) [8].

$$3Mg^{2+} + 6OH^{-} + 4SiO_2 \rightarrow Mg_3Si_4O_{10}(OH)_2 + 2H_2O \quad (1)$$

MSH (Talc) improves the fracture properties of the surface by converting adhesion fractures into cohesion fractures. It has a high resistance to heat, electricity, and acid due to its flake structure. Talc is widely used as a functional pigment such as paints, ceramics, and cosmetics due to its high mechanical strength, chip resistance, color effects, insoluble in water, and corrosion resistance [9,10]. Lefebvre et al. have modified the surface of Talc layers by dry coating method with different level of hydrophobic Silica. They have controlled the wettability and dispersity of particles in aqueous solutions with modifying the surface of Talc particles. [10]. Du et al. (2008) prepared the three and four-layer structure pigment systems by the conventional wet chemical method. These functional pigments were prepared by precipitating different metal oxide ions [11].

Recently, many studies have reported the syntheses of pearlescent pigments and characterized their optic properties [12-14]. But there is a lack of studies on dielectric properties of special pigments. In this study, a novel approach for applications of MSH pigments was discussed due to their high dielectric properties. The SnO_2 deposited MSH pigments were synthesized by the chemical reduction method. The dielectric properties of these pigments were investigated in the frequency range of 8-12 GHz.

2. MATERILAS AND METHODS

The MSH (Talc) flakes with the particle size ranging 10-50 μm was provided by ISIK Madencilik in Turkey. These MSH flakes were milled and then passed through a 25 μm sieve for uniform and smaller size distribution. SnO_2 and $NaBH_4$ nanoparticles were purchased from Sigma Aldrich supplier.

The SnO_2 nanoparticles with 20-50 nm were deposited on MSH flakes via chemical reduction method. The SnO_2 ratio to MSH is 3:7 by weight. Firstly, MSH particles were mixed in pure water under magnetic stirring for 2h. Secondly, SnO_2 particles were added to the MSH solution and keep stirring 2 h. 93 mg $NaBH_4$ was dissolved in 3 ml pure water mechanically. Finally, $NaBH_4$ solution was added to the MSH solution and stirred 24 h. The solution was centrifuged and dried at 80°C. The MSH/SnO_2 and pure MSH were labeled as T1 and T2 respectively. The surface structure of obtained pigments was obtained by using field-emission scanning electron microscope (FESEM) and phases properties of pigments were determined by Rigagu ULTRA IV x-ray diffraction (XRD). The bond type of particles was characterized by Bruker ALPHA Fouirer Transform Infrared Spectrophotometer (FTIR) spectrometer. For dielectric properties, the obtained particles were added to epoxy with 3:7 mass ratio and dielectric properties were determined by using a two-port waveguide technique with a vector network analyzer.

3. RESULTS AND DISCUSIONS

The SEM images of MSH (T2) and SnO_2 (T1) deposited MSH pigments are given in figure 1. The surface of talc flakes detected as a smooth lamella-shaped with 2-10 μm sizes. The surface of MSH pigments was coated uniformly with SnO_2 nanoparticles. It was proved that the chemical reduction is a suitable method for doped SnO_2 on MSH flakes.

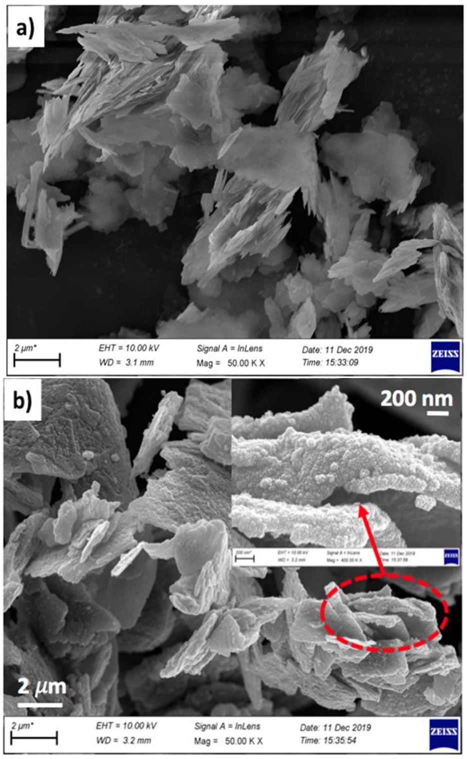

Figure 1. SEM images of MSH (a) and SnO_2 deposited MSH

Figure 2 shows the FT-IR spectra of T1 and T2 pigments. The typical peaks at 668 cm^{-1} correspond to the stretching of Si–O–Si bending. Another peak that appears around 1013 cm^{-1} was assigned to Si-O vibration. Nied et al. have reported that the intensity of the 1013 cm^{-1} peak depends on the Mg/Si ratio [15]. They have obtained that the peak intensity has increased with an increase of Mg/Si ratio. The OH peak from brucite (MgOH) was characterized by the sharp band at 3675 cm^{-1} [7,15].

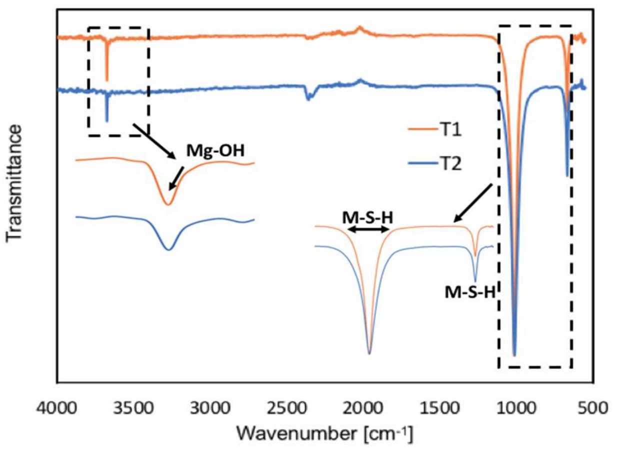

Figure 2. FT-IR spectra of T1 and T2

XRD result of MSH (3*a*) and MSH/SnO_2 (3*b*) presented in Fig. 3. The peaks at 9,5, 19, 29, 37, 49, 61° 2θ is peaks of Talc ($Mg_3Si_4O_{10}(OH)_2$). SiO_2 peaks which are another compound of MSH are detected at 27 and 32° [16]. The intensity of peaks obtained at 9,5 and 29 decreased after SnO_2 deposition on MSH. The tetragonal SnO_2 reflections were obtained at peaks (110), (101), (211), (211), (220) and (002) in 28.7, 52.3, 55,4 and 58,7° respectively [6].

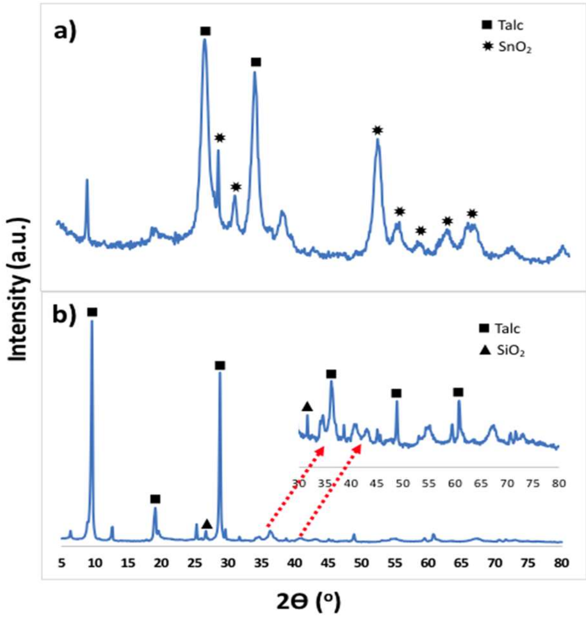

Figure 3. XRD patterns of T1 and T2

The dielectric constants of the T1 and T2 (reel (ε') and imaginary (ε'') part of permittivity) are given in Figure 4*a,b*. For T1, two decreasing peaks have appeared from ε' while two increasing peaks obtained from ε'' at the same frequency range. As seen in figure 4a and 4b, the dielectric constants (reel and imaginary) of T2 (MSH) are relatively stable but dielectric properties of T2 (SnO_2 deposited MSH) was obtained to be about almost 3-4 times than those of obtained from T1 in the 8-12 GHz frequency range. These are can be attributed to interfacial polarization [17-19]. Zhu et al. have attributed this to the existence of dipoles moments resulting from the natural physical properties of the shell. [20]. It was clearly seen that the deposition of SnO_2 has increased the dielectric constant of MSH.

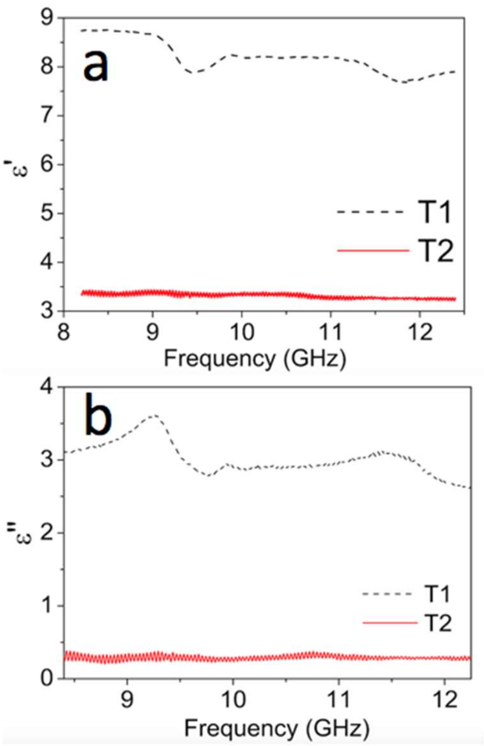

Figure 4. Frequency dependence of dielectric properties of T1 and T2, real permittivity **a**, and imaginary permittivity **b**.

The dielectric loss tangent (tan $\delta\varepsilon = \varepsilon''/\varepsilon'$) of T1 and T2 were calculated to evaluate microwave attenuate performance and given in Fig. 5. It can be clearly seen that SnO_2 deposited MSH pigment (T1) provided higher dielectric loss tangent values. The dielectric loss tangent value of T1 reached a peak value of 0.45 between 9-10 GHz. It was concluded that the deposition of SnO_2 to talc flake gained high dielectric loss and microwave attenuate performance.

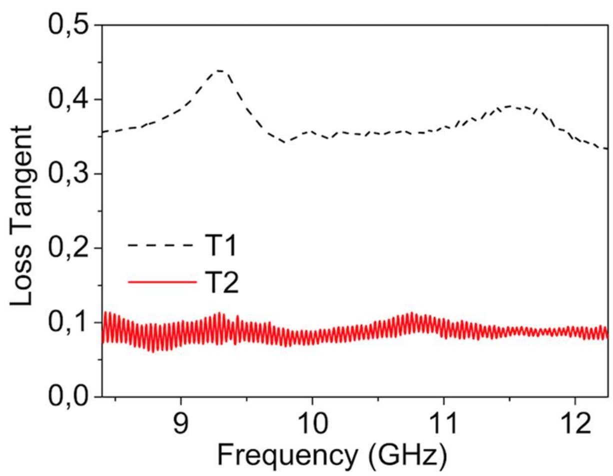

Figure 5. Dielectric loss tangent of T1 and T2

4. CONCLUSION

The MSH/SnO_2 pigments were synthesized by chemical reduction method. The structural and dielectric characterizations were carried out. The existence of SnO_2 on MSH flakes was proved by SEM and XRD results. The SEM images showed that SnO_2 nanoparticles were deposited on surface of MSH continuously. The existence of SnO_2 increased the reel (ε') and imaginary (ε'') part of dielectric values. In addition, the higher dielectric loss tangent values were obtained with SnO_2 coated. This study presents a novel approach for applications of MSH pigments due to their high dielectric and loss properties.

Acknowledgments

This study is supported by Van YYU University Scientific Research Projects Coordination Unit. Project Number: FBA-2019-7959.

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