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ELECTROMAGNETIC SHIELDING PERFORMANCES OF COLEMANITE / PANI / SIO2 COMPOSITES IN RADAR AND WIDER FREQUENCY RANGES

Ethem İlhan ŞAHİN*, Mehriban EMEK, Burcu ERTUĞ***, Mesut KARTAL******

ABSTRACT

In this study, colemanite-SiO2 were produced by using mixed oxide technique. The composition was formed with various proportions for the structural analysis. The results of the structural analysis indicated that second phase did not form in colemanite and SiO2. Addionality, the colemanite/polyaniline/SiO2 composites were produced by hot pressing using the compositions of colemanite-SiO2 in different proportions and aniline. The weight ratios of colemanite-SiO2 and aniline were 1:1 respectively and epoxy resin was used to produce microwave shielding composites. The microwave shielding performances of colemanite/polyaniline/SiO2 composites were investigated by shielding effectiveness in 8 –18 GHz using two–port vector network analyzer. A minimum of – 41.1 dB shielding effectiveness performance was obtained in 16.09 GHz at the thickness of 1.5 mm. This shielding performance can be modulated simply by controlling the content of polyaniline and content of colemanite-SiO2 in the samples for the required frequency bands.

Keywords: *Elektromagnetic shielding effectiveness, polyaniline, colemanite, polymer-matrix composites.*

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RADAR VE DAHA GENİŞ FREKANS ARALIĞINDA KOLEMANİT/PANI/SiO2 KOMPOZİTLERİN ELEKTROMANYETİK KALKANLAMA PERFORMANSLARI

Ethem İlhan ŞAHİN*, Mehriban EMEK**, Burcu ERTUĞ***, Mesut KARTAL****

ÖZ

Bu çalışmada, kolemanit-SiO_2 oksitlerin karışımı tekniği kullanılarak üretilmiştir. Kompozisyon, yapısal analiz için çeşitli oranlarda oluşturuldu. Yapısal analizin sonuçları, kolemanit ve SiO_2'de ikinci fazın oluşmadığını göstermiştir. Buna ek olarak, kolemanit /polianilin/SiO_2 kompozitleri, farklı oranlarda kolemanit-SiO_2 bileşimleri ve anilin kullanılarak sıcak presleme yoluyla üretildi. Kolemanit-SiO_2 ve anilin ağırlık oranları sırasıyla 1: 1 idi ve mikrodalga koruyucu kompozitler üretmek için epoksi reçine kullanıldı. Kolemanit/polianilin/SiO_2 kompozitlerinin mikrodalga kalkanlama performansları, iki portlu vektör network analizörü kullanılarak 8-18 GHz'de ekranlama etkinliği ile araştırılmıştır. Minimum -41.1 dB ekranlama etkinliği performansı 1.5 mm kalınlığında 16.09 GHz'de elde edilmiştir. Bu ekranlama performansı, gerekli frekans bantları için numunelerde polianilin içeriği ve kolemanit-SiO_2 içeriği kontrol edilerek modüle edilebilir.

Anahtar Kelimeler: *Elektromanyetik kalkanlama etkisi , polianilin , kolemanit, polimer-matrix kompozitler.*

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1. Introduction

Due to the extensive use of modern communication gadgets,Electromagnetic Interference (EMI) has received worldwide social and scientific attention. In general,critical EMI problems are encountered in satellite communication, digatial devices, radar management systems, wireless technology and other electronic equipment [1-3]. Due to the intense generation of high frequency electronic industry, electromagnetic pollution and electromagnetic interference (EMI) have emerged as a major issue in today's world. Electromagnetic radiation not only impedes electrical power system efficiency and output but also has a damaging influence on human health. Using EMI shielding materials is an useful strategy to reduce the above mentioned problems efficiently. In just this regard, new and more effective EMI shielding materials have attracted significant attention [4-8]. Shielding of EM waves leads to reduction of interferences by converting EM energy into thermal energy via reflection or absorption of radiation power [9]. For instance, Metals and carbon materials are fine EMI shielding materials [10-13].

Polymer-based electrical conductor composites offer additional benefits over conventional metallic composites, including cost-effective, lightweight and corrosion-resistant [14].

Among the intrinsically conductive polymers, the extraordinary electrical property of polyaniline (PANI) coupled with its low density and process-efficiency attracted great interest as a new functional component to produce multifunctional composites [15-17].

Under controlled environments, PANI can be produced by aniline's chemical oxidative polymerization. PANI can also be doped easily and shows sufficient stability [18-20].Recent work it has been shown that by implementing heterostructure, the shielding capability of PANI-based composites can be noticeably improved [21].

Colemanite is a large variety of boron mineral types in the earth; $CaO \cdot 3B_2O_3 \cdot 5H_2O$(colemanite) is the most widely used as commercial boron minerals in the world [22].They are also very effective material as boron is commonly used in many fields, which include glass, agriculture, detergent, wood beauty products, clothing, cloth, rubber, paint Besides , boron is innovative application areas for both the nuclear, energy storage, ceramic and materials science industries [23]. For two principal reasons, SiO2 is the most commonly used inorganic support. Firstly, in the literature the formation of monodispersed silica particles is really quite easy to execute and well recorded [24]. Second, different potential applications of silica-based systems are associated: gas-liquid chromatography, supports to catalysts, etc.[25].Single polymer membrane separators, however, can not fulfill the standard for better mechanical properties and electrochemical properties at about the same time, so compounding various polymers[26–28] or connecting other compounds such as CuO[29] and **SiO_2**[30-32] to the polymer matrix is the most effective means of improving the separator effectiveness.

In previous studies in the literature, the microwave EMI shielding properties of polyaniline composite with Ag decorated (5.0 wt. % loading) graphene was reported to be 29.33 dB [33]. In addition, Shuong et al. recorded required parameters with an EMI shielding of ~30 dB in Cu / Ni coated fiber, developed by electroless technique [34].

The magnitude of the shielding effect is connected to how far the incoming electromagnetic wave is going through. For the shielding value of -10 dB, it is understood that the incoming electromagnetic wave is reduced by 90 per cent and that the opposite side is passed by 10 per cent. [35-36].

In this study the single phases colemanite-SiO2 were prepared for the first time by means of mixing oxides as solid solution in different ratios. Then new colemanite-SiO2: PANI were produced using this composition.

2. EXPERIMENTAL SECTION

2.1 Preparation of colemanite-SiO2

Colemanite-SiO2 powders were produced using mixed oxide technique. Ground colemanite mineral (GC) obtainable as an commercial product is made and offered for sale by the Company (Eti Mining Company, Turkey) in some of these compositions with particulate matter of -75 μm and 10 nm silicon dioxide (SiO2) (Sigma–Aldrich: 99 %) powders were mixed in stoichiometric amounts, according to the colemanite-SiO2 compositions in the ethanol medium, it was mixed for 20 hours in ethanol medium in a plastic container at 25-75 wt. % , 50-50 wt. % and 75-25 wt. %, respectively. After the slurries were dried at 100 °C for 24 h, they were calcined at 600 °C for 4 h in a tightly closed alumina crucible to prevent losses of evaporation, which were tested by weighing the samples before and after calcination. . They were pressed into pellets with a diameter of 10 mm and a thickness of 1 – 2 mm using uniaxial press with 2 MPa pressure, after the calcined powders were ground in an agate mortar. The pellets were sintered between the 900 °C for 4 h with a heating and cooling rate of 250 °C/h after having buried them in the colemanite-SiO2 powders to minimize the loss of volatile species. The single-phases colemanite-SiO2 reactant powders were sintered at 900 °C after being calcined at 600 °C.

The phases in sintered samples were characterized by X-ray diffractometry (XRD- D2 Phaser Bruker AXS) with Cu- Kα radiation (λ = 1.5406 Å) in the range 2θ:10 – 70° at a scan rate of 1°/min. The solubility limit, which is defined as the amount that can be doped without disturbing the structure of the main structure (colemanite-SiO2), was determined using X-ray powder diffractometry. The microwave shielding effectiveness performances of the colemanite/PANI/ SiO2 composites were measured with the two-port vector network analyzer (R & S FSH-K42) device (Fig. 1) in the range of 8 – 18 GHz.

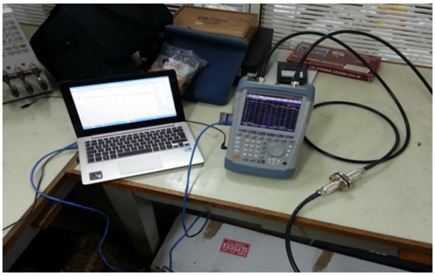

Figure 1. Two-port vector network analyzer (R & S FSH-K42) device (8-18 GHz)

2.2. Preparation of polyaniline/colemanite-SiO2 composites

The single-phases colemanite-SiO2 (at 25-75 wt. %, 50-50 wt. % and 75-25 wt. %, res $CaO \cdot 3B_2O_3 \cdot 5H_2O$ which have the compositions of CaO·3B2O3·5H2O and SiO2 (account for 100 wt.% of aniline quantity), and 1 ml aniline monomer were added in 35 ml hydrochloric acid solution (0.1 mol L-1) and dispersed by mechanical stirring for 30 min. 2.49 g of ammonium persulfate (APS) was dissolved in a 15 ml hydrochloric acid solution (1 mol L-1). The APS solution was then carefully added by stirring vigorously dropwise to the preceding mixture solution. Polymerization was done at 0°C for 12 h in an ice-water bath. The composites were produced by filtering and washing the reaction mixture with deionized water and ethanol, which was then vacuum-dried for 24 hours at 60 °C. The PANI/ colemanite-SiO2 composites with different molar ratios [(Aniline/ colemanite-SiO2 (at 25-75 wt.%), Aniline/ colemanite-SiO2 (at 50-50 wt. %), and Aniline/ colemanite-SiO2 (at 75-25 wt. %) = (1:1)] were obtained to investigate the influence of the PANI content on the electromagnetic shielding effect properties. The colemanite-SiO2 compositions were produced as composite with a PANI base. PANI- colemanite: SiO2 was produced by hot pressing at different ratios.

2.3. Preparation of epoxy-polyaniline/ colemanite : SiO2 composites

The composite materials were made ready by molding and curing the mixture of PANI / colemanite: SiO2 compositions powders and epoxy. The specimen powder to epoxy mixing ratio was 2:1 by weight. At 5 MPa pressure and 100 °C for 1 h, the molding was carried out in a hydraulic press.
They were pressed into pellets with a 20 mm diameter and 2 mm thickness for shielding measurements. Composites of microwave shielding Effectiveness were produced using epoxy at different ratios of aniline / colemanite: SiO2 such as 1/1.

3. RESULTS AND DISCUSSION

3.1. XRD analysis of colemanite-SiO2 composites

To define the mineralogical characterization of ground colemanite, XRD device diffracted its X-ray pattern. The XRD analysis of the samples (colemanite, SiO2) sintered at 900 °C for 4 h revealed that single phase structure was formed , additionally PANI was analyzed using XRD device (Fig. 2). As could be seen on the detecting of colemanite, SiO2 and PANI XRD patterns (Fig. 2), main phases are determined as Colemanite (PDF Card No: 01-082-1825), SiO2(PDF Card No :01-077-1060) , Polyaniline (PDF Card No :00-053-1717). The single phase structure of the powders was achieved while using the mixed oxide synthesis with a suitable calcination temperature and elimination of the possible intermediate phases. The more well homogenization of the powders during heating improved the diffusion process.
The XRD tests showed that there was no secondary phase in the powders for colemanite, polyaniline and SiO2. Furthermore, colemanite-SiO2 formation largely depends on temperature and high temperatures are sometimes required to form single phases.

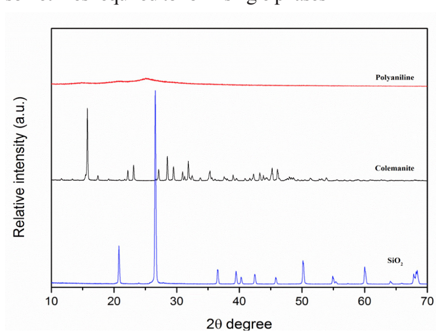

Figure 2. XRD patterns of SiO2, Colemanite (for 4 h at 900°C), PANI compositions

3.1. EMI Shielding Measurements of colemanite/PANI/SiO2 composites

Figure 3 shows the frequency dependence of the shielding effect of the Epoxy- PANI/colemanite: SiO2 composites in the frequency range of 8-18 GHz. Among the PANI- colemanite: SiO2 composites, It can be seen that epoxy- colemanite: SiO2 (50-50 wt. %) composites /Aniline: 1/1 has more observable effect on microwave shielding effectiveness properties than other composites. The epoxy-PANI/colemanite:SiO2 compositions (colemanite:SiO2 (25-75 wt. %) /Aniline: 1/1) powders and epoxy showed only one band with -32.32 dB at 17.52 GHz. In addition, this composite material achieves a shielding effect less than -10 dB in the frequency band between 8 GHz and 18 GHz. Morever, it achieved a shielding effect less than -15 dB in the frequency bands between 8 GHz and 16.65 GHz. Besides ,it achieved a shielding effect less than -20 dB in the frequency bands between 8 GHz and 9.32 GHz, 15.53 GHz and 16.17 GHz (Fig3.a). When powder content is equal, the epoxy-PANI/ colemanite:SiO2 compositions (colemanite:SiO2 (50-50 wt. %) /Aniline: 1/1) reach to -41.10 dB and -31.24 dB, at 16.09 GHz and 17.70 GHz, respectively (Fig3.b). Moreover, it achieved a shielding effect less than – 10 dB in the frequency bands between 8 GHz and 18 GHz. Morever, it achieved a shielding effect less than -15 dB in the frequency bands between 8 GHz and 16.65 GHz. Besides, it achieved a shielding effect less than -20 dB in the frequency bands between 14.04 GHz and 16.61 GHz. The compositions reach to a shielding effectiveness of -31.62 dB at 17.52 GHz for epoxy-PANI/ colemanite:SiO2 compositions (colemanite:SiO2 (75-25 wt. %) /Aniline: 1/1) (Fig3.c). Moreover, it achieved a shielding effect less than – 10 dB in the frequency bands between 8 and 16.65 GHz, 17.37 GHz and 18 GHz, respectively.
Morever, it achieved a shielding effect less than -15 dB in the frequency bands between 8 GHz and 9.37 GHz, 14.82 GHz and 16.61.
Impedance matching was achieved through the use of PANI. PANI has effected the shielding effectiveness. Additionally, PANI and composites play a great role in the efficacy of the electromagnetic shielding material. The efficacy of micro-wave shielding also depends on matching the impedance of irradiation to the material surface. PANI enhances the matching impedance of connections between the composite components.
New epoxy-PANI/colemanite:SiO2 compositions were produced, colemanite:SiO2/ PANI composite has a high shielding effect ratio for electromagnetic waves in a broadwidth range, Microwave shielding effect of new composites are adjusted by regulating the content of colemanite:SiO2 and PANI in this process. Studies indicate that the content of colemanite:SiO2 and PANI affect structure.

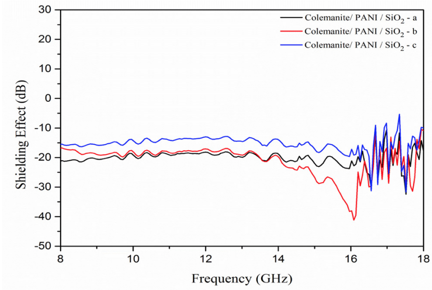

Figure 3. Microwave shielding effectiveness of the epoxy- PANI/colemanite:SiO2 composites: a – colemanite:SiO2 compositions (colemanite:SiO2 (25-75 wt. %) /Aniline: 1/1, b – colemanite:SiO2 compositions (colemanite:SiO2 (50-50 wt. %) /Aniline: 1/1 c – colemanite:SiO2 compositions (colemanite:SiO2 (75-25 wt. %) /Aniline: 1/1.

4. Conclusion

Colemanite-SiO2 powders (at 25-75 wt. %, 50-50 wt. % and 75-25 wt. %, respectively) were produced by using solid state technique and PANI/colemanite:SiO2 composites were produced for the first time in literature, to our knowledge.

The microwave shielding effectiveness property was obtained as 16.09 GHz and 1.5 mm in thickness with the minimum SE of -41.10 dB by the epoxy-PANI/ colemanite:SiO2 compositions (colemanite:SiO2 (50-50 wt. %) /Aniline: 1/1). Microwave shielding properties can be modulated simply by regulating the content of PANI and the effect of colemanite:SiO2 content on the samples for the required frequency bands. Due to the easy and low cost preparation methods and better shielding effectiveness performance, the PANI / colemanite:SiO2 composites have a promising potential as microwave shielding effectiveness. Content of colemanite:SiO2 and Polyaniline were used to improve the microwave shielding effectiveness.

Microwave shielding properties of PANI / colemanite:SiO2 compositions show a strong variability with high concentrations of colemanite:SiO2 content. . The best shielding effect performance is obtained from colemanite:SiO2 (50-50 wt. %) / Aniline: 1/1 composition at the value of less than -20 dB and between 14.04 GHz and 16.61 GHz.

1/1 composition at the value of less than -20 dB and between 14.04 GHz and 16.61 GHz. The second shielding effect performances are obtained from colemanite:SiO2 (50-50 wt. %) /Aniline: 1/1 and colemanite:SiO2 (25-75 wt. %) /Aniline: 1/1 compositions at the value of less than -10 dB and between 8 GHz and 18 GHz.

The polyaniline content plays an significant role in variation of the shielding effectiveness. The microwave shielding effect properties of the PANI / colemanite:SiO2 compositions can be investigated for a broader concentration range in this study. PANI / colemanite:SiO2 can be considered as a candidate for microwave shielding effectiveness in a broadwidth range. The formation of colemanite:SiO2 content is another area. Colemanite:SiO2 and PANI content have been used to improve the microwave shielding properties. The microwave shielding effectiveness properties of PANI - colemanite:SiO2 at low frequencies may also be determined.

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