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Research Article

Electromagnetic Shielding Effectiveness of Wollastonite/PANI/Colemanite Composites

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Abstract

In this study, composites of wollastonite-colemanite were produced by using mixed oxide technique. The wollastonite-colemanite compositions were formed with various proportions for the structural analysis. The results of wollastonite-colemanite structural analysis indicated that second phase did not form in wollastonite and colemanite. The single phases wollastanite-colemanite compounds were measured after sintering between 900-1100°C for X-ray diffraction (XRD). Addionality, the wollastonite/polyaniline/colemanite composites were produced by hot pressing using the compositions of wollastonite-colemanite in different proportions and aniline. The weight ratios of (wollastonite-colemanite) and aniline were 1:1 respectively and epoxy resin was used to produce microwave shielding effectiveness composites. The microwave shielding performances of wollastonite/polyaniline/colemanite composites were investigated by shielding effect in 0 –8 GHz, using two–port vector network analyzer (VNA). A minimum of – 41.65 dB shielding effectiveness performance was obtained in 6.26 GHz at the thickness of 1.5 mm. According to the parameters determined in terms of properties, the wollastonite-colemanite compounds were produced as composite with a PANI base and their features were characterized for shielding effect. This microwave shielding performance can be modulated simply by controlling the content of polyaniline and content of wollastonite-colemanite in the samples for the wider and required frequency bands.

Keywords: Electromagnetic shielding effectiveness, Polyaniline, Wollastonite, Colemanite, Polymer-matrix composites.

Wollastanit/PANI/Kolemanit Kompozitlerin Elektromanyetik Kalkanlama Etkinliği

Öz

Bu çalışmada, wollastanit-kolemanit kompozitleri oksitlerin karışımı tekniği kullanılarak üretilmiştir. Wollastanit-kolemanit bileşimleri, yapısal analiz için çeşitli oranlarda oluşturulmuştur. Wollastonit-kolemanit yapısal analiz sonuçları, wollastanit ve kolemanit'te ikinci fazın oluşmadığını göstermiştir. Tek fazlı wollastanite-kolemanit bileşikleri, X-ışını kırınımı (XRD) için 900-1100°C arasında sinterlendikten sonra ölçüldü. Ayrıca wollastanit /polianilin/ kolemanit kompozitleri, farklı oranlarda wollastanit-kolemanit ve anilin bileşimleri kullanılarak sıcak presleme ile üretilmiştir. (Wollastonite-Kolemanit) ve anilinin ağırlık oranları sırasıyla 1:1 idi ve epoksi reçinesi mikrodalga kalkanlama etkili kompozitleri üretmek için kullanıldı. Wollastanit/ polianilin / kolemanit kompozitlerinin mikrodalga kalkanlama performansları, iki portlu vektör network analizörü (VNA) kullanılarak 0–8 GHz' de ekranlama etkisi ile incelenmiştir. 1.5 mm kalınlıkta 6.26 GHz'de minimum - 41.65 dB ekranlama etkinliği performansı elde edildi. Özellikler açısından belirlenen parametrelere göre wollastanit-kolemanit bileşikleri PANI bazlı kompozit olarak üretilmiş ve özellikleri kalkanlama etkisi için karakterize edilmiştir. Bu mikrodalga kalkanlama performansı, daha geniş ve gerekli frekans bantları için numunelerdeki polianilin içeriği ve wollastanit-kolemanit içeriği kontrol edilerek kolay bir şekilde modüle edilebilir.

Anahtar Kelimeler: Elektromanyetik kalkanlama etkinliği, Polianilin, Wollastanit, Kolemanit, Polimer-matrisli kompozitler.

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1. Introduction

Particularly with the recent development of electronic equipment and gadgets that emit electromagnetic energy in various frequency ranges, Protecting digital devices against causes of interference has become critical [1]. By causing data leakage, mistaken operations, or perhaps even total failure, EMI may affect the functionality of electronics devices. Also there is an increasing concern more about harmful effects on human health of electromagnetic (EM) radiation, particularly from future 5th generation (5 G) communication systems [2-3]. There is a serious impact of electromagnetic radiation on people's health. That use EMI shielding materials is a powerful technique to significantly reduce the above problems in an effective method. Novel and efficient EMI shielding materials have received considerable attention in this respect [4-6].

In particular, the best shielding material must be with properties as with electrical conductivity, excellent thermal, and high EMI shielding efficiency [7-9]. Overall, in recent times, such features involving EMI shielding materials have been in great demand. EM radiation absorption and reflection are two main mechanisms of EMI shielding, resulted from its direct interaction of mobile charge carriers and magnetic / electric dipoles, respectively, in shielding materials with EM fields. The materials' electrical conductivity is known to be the most a fundamental parameter for controlling the characteristics of EMI shielding [2,3,10].

To EMI shielding, composite materials with discontinuous conducting filler including such metal flakes, metallic wires and particles and carbon fibers are commonly used[11]. With in past few years, due to its excellent electrical conductivity, metal-based composites have attracted a great deal of attention.[10,12-13] In addition, in the gigahertz frequency region, they have even been potentially used as EMI shielding materials [14]. In addition, due to its specific characteristics such as high conductivity and tunable EMI shielding performance, conductive polymer composites are great candidates for EMI shielding [15-16]. Polymer-based electrical conductor composites provide additional benefits such as lightweight, cost-effective and corrosion-resistant composites, compared to traditional metallic composite materials [17]. In order to acquire polymer composites with shielding properties, there are two common tactics. The first is the incorporation of functional conductive fillers into the matrix of the insulating polymer [18-20]. In order to achive enhanced EMI shielding, the creation of a well-established conductive network is necessary. High intrinsic conductivity and/or high loading of functional fillers are usually needed to achieve acceptable shielding efficiency [21-22]. Other approach is to apply the coating technique by utilizing layered structure to produce polymer composites. Because the functional components appear to be limited to a thin coating sheet, the local conductive component concentration is increased. In this system, the electrical conductivity networks are rapidly created. By this efficient and simple strategy, the shielding efficiency of composites can be improved effectively [23-24].

Due to its remarkable properties, such as good electrical conductivity and non-transparency to EM radiation, conductive polymers such as polyaniline (PANI) [25-26], polypyrrole,[27] and poly(3,4-ethylene dioxythiophene) are also reported to be beneficial in increasing EMI shielding efficiency. The specific l physical andchemica properties of polyaniline (PANI) are known as, good environmental stability, low-cost and ease of production [28]. Compounds made of polymers and inorganic / organic filling materials are polymer composites. In controlled environments, PANI can be manufactured by chemically oxidative polymerization of aniline. PANI can be indeed doped easily and shows adequate stability [29-31].

The natural calcium metasilicate ($CaSiO_3$) is wollastonite. Technically, wollastonite contains 48.3 wt. % CaO and wt. % 51.7 SiO_2. It is seldom found to be pure and occurs as large-leaved masses. It is commonly observed with a fibers framework in sharp-looking or tiny particles such as needles [32]. Wollastonit has high temperature resistance and high mechanical resistance. It is possible to control its porosity, it can be easily pressed and its insulating capability is good. Wollastonit is a raw material which is non-carcinogenic, readily available and transportable [33-34]. Wollastonite is very known inorganic filling used in plastics that occurs naturally, which improves mechanical properties such as stiffness and tensile strength but decreases the strength of the impact. With the application of a proper elastomer as an impact modifier, which can also serve as a compatibilizer, this limitation can be eliminated. Wollastonite has been used as a reinforcing for epoxy resin, polystyrene and PVC [35-36].

One of most significant boron minerals is Colemanite ($Ca_2B_6O_{11}.5H_2O$). The concrete samples obtained in the concrete mixture using colemanite can become an effective armor for gamma and neutrons radioactivity. This mineral is used in nuclear reactor for power bars [37]. Colemanite is the most commonly used industrial boron mineral in the earth [38]. They are very influential material since boron is widely used in a great deal of fields, including detergent, glass, agriculture, cosmetics, leather, rubber, paint. In addition to, boron is a revolutionary application field for both nuclear energy, ceramics, energy storage [39].

Besides, single polymer membrane separators do not meet the norm for better electrochemical properties and mechanical properties almost at the same time, because the most efficient way to enhance the effectiveness of the separator is to compound different polymersor add other compounds to the polymer matrix [40-41].

In previous research, the Colemanite / Pani / SiO_2 composite has quite a sample width of 1.5 mm and a maximum electromagnetic shielding effect of -41.1 dB at 16.09 GHz frequency [42]. Morever, an economical box-test setup has been developed to be used for textile composite with electromagnetic shielding effectiveness research [43]. Electromagnetic shielding properties of wood composites coated with vermiculite doped rigid polyurethane were also examined [44]. In another analysis, for instance, the maximum electromagnetic shielding effect were calculated as-39 dB at 1.6 GHz for 0.25 wt % MWCNT composites [45].

The performance value of the shielding effect is connected to how far the incoming electromagnetic wave passes through the material. It is known that the incoming electromagnetic wave is decreased by 90 percent with a shielding effect value of -10 dB, and 10 percent passes to the opposite side [46-47].

The wollastonite-colemanite compounds were produced as a composite with a PANI base according to the optimal parameters defined in terms of properties, and their shielding effect characteristics were characterized. (Wollastonite-colemanite):PANI was manufactured at various proportions by hot pressing. Composites of microwave shielding effectiveness were produced using epoxy at various proportions of aniline / (wollastonite-colemanite) compound such as 1/1. New (wollastonite-colemanite):PANI composites were produced. XRD (Bruker / Alpha-T) devices were used to identify the composites that were characterized. The microwave shielding effect values of (Wollastonite-colemanite):PANI composites were calculated in the frequency range of 0-8 GHz, including wlan and mobile phone frequencies, by the two-port vector network analyzer (R & S FSH-K42) machine.

2. Material and Method

2.1. Preparation of Wollastonite-Colemanite

Wollastonite-colemanite powders were obtained as a compound with using mixed oxide technique. Wollastonite powders, which can be purchased as a commercial product, are manufactured and offered for sale by the company (Tecnodieci Company, Eskişehir, Turkey) in some of these 40 μm particle size compositions. Wollastonite powders, which can be purchased as a commercial product, are manufactured and offered for sale by the firm (Tecnodieci Company, Eskişehir, Turkey) in some of these 40 μm particulate size compositions. Wollastonite powders, which can be purchased as a commercial product, are manufactured and offered for sale by the firm (Tecnodieci Company, Eskişehir, Turkey) in some of these 40 μm particulate size compositions.

Ground colemanite mineral (GC) reachable as a commercial product, in some of these compositions with a particle size of 75 μm, is produced and offered for sale by a company (Eti Mining Company, Turkey). Colemanite and Wollastonite powders were mixed in stoichiometric quantities, according to the wollastonite-colemanite compositions in the ethanol medium, powders were mixed for 20 hours in ethanol medium in a plastic bottle at 25-75 wt. %, 50-50 wt. % and 75-25 wt. %, respectively.

After the slurries were dried at a temperature of 100 °C for 24 h, they were calcinated at a temperature of 600 °C for 4 h in a fully closed alumina crucible to avoid evaporation losses, which were checked before and after calcination by weighing the samples. Using a uniaxial press with 2 MPa pressure, they were pressed into pellets with just a diameter of 10 mm and a thickness of 1-2 mm, after the calcinated powders were grounded in an agate mortar. After being mounted in a sealed crucible, the pellets were sintered at 1000 °C for 4 hours at a heating and cooling rate of 300 °C /hour to reduce the loss of volatile species. Powders of single phase wollastonite-colemanite were calcinated at 600 °C and then sintered at 1000 °C. The phases in sintered samples were characterized by XRD (X-ray diffractometry - D2 Phaser Bruker AXS) with Cu- Kα radiation (λ = 1.5406 Ǻ) in the range 2θ:10 – 70° and at a scan rate of 1°/min. Using X-ray powder diffractometry, the solubility limit, described as without disturbing the structure of the main structure (wollastonite-colemanite), was determined. The microwave shielding effectiveness of the wollastonite/ PANI /colemanite composites were determined in the frequency range of 0-8 GHz with the two-port vector network analyzer (R & S FSH-K42).

2.2. Preparation of Polyaniline / (Wollastonite-Colemanite) Composites

The single-phases wollastonite-colemanite (at 25-75 wt. %, 50-50 wt. % and 75-25 wt. %, respectively) powders which have the compositions of $CaSiO_3$ and ($Ca_2B_6O_{11}.5H_2O$) (account for 100 wt. % of aniline quantity), and 1 ml aniline monomer were applied to 35 ml hydrochloric acid solution (0.1 mol L-1) and dispersed for 30 min. by mechanical stirring. 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 slowly activated by stirring strongly dropwise to the preceding mixture solution. Polymerization was completed at 0°C for 12 h in an ice-water bath. The composites were manufactured by filtering and washing the reaction mixture with deionized water, and ethanol, which was then vacuum-dried for 24 h at 60 °C. The PANI/ wollastonite- colemanite composites with different ratios [(Aniline/ wollastonite- colemanite (at 25-75 wt.%), Aniline/ wollastonite- colemanite (at 50-50 wt. %), and Aniline/ wollastonite- colemanite (at 75-25 wt. %) = (1:1)] were produced to investigate the effect of the PANI ingredient on the electromagnetic shielding effect characteristics. The wollastonite-colemanite compositions were obtained as a PANI based composite. PANI/ wollastonite- colemanite was fabricated at different ratios by hot pressing.

2.3. Preparation of Epoxy- (Polyaniline / Wollastonite–Colemanite) Composites

The composite materials were prepared via molding and curing the mixture of powders and epoxy compositions of (wollastonite-colemanite) / PANI. 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. For shielding effectiveness calculations, they were pressed into pellets with a 20 mm diameter and 1.5 mm thickness. Composites of microwave shielding effectiveness were fabricated using epoxy at particular of aniline / (wollastonite-colemanite) as in 1/1.

3. Results and Discussion

3.1. XRD Analysis of Wollastonite-Colemanite Composites

The XRD device diffracted its X-ray pattern to describe the mineralogical characterization of wollastonite and colemanite. The XRD analysis of the samples (wollastonite, colemanite) annealed at 1000 °C for 4 h demonstrated that single phase structure was formed, additionally PANI was analyzed utilizing XRD device (Fig. 1). As could be seen on the detecting of wollastonite, colemanite and polyaniline XRD patterns (Fig. 1), pricipal phases are defined as wollastonite (PDF Card No: 00-042-0550), colemanite (PDF Card No: 01-082-1825), polyaniline (PDF Card No: 00-053-1717). The single phase structure of the powders was obtained while using the mixed oxide synthesis with a suitable calcination temperature and removal of the potential intermediate phases. The more well homogenization of the powders during heating enhanced the diffusion process. All of the samples were sintered at 1000 ºC for 24 hours. The XRD analysis revealed that there was no secondary phase in the powders for wollastonite, colemanite and PANI. The diffraction peaks of the samples were consistent with wollastonite, colemanite and their phase structures were still pure wollastonite-colemanite phase. In addition, wollastonite-colemanite formation mainly counts on temperature and high temperatures are sometimes needed to form single phases.

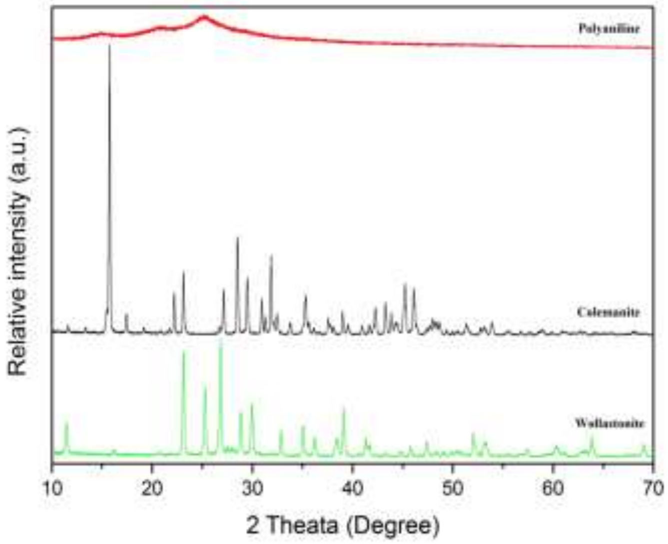

Figure 1. XRD patterns of wollastonite, colemanite and PANI compositions, sintered (only wollastonite and colemanite) during 4 h at 1000°C

3.2. EMI Shielding Measurements of Wollastonite/PANI/Colemanite Composites

Figure 2 shows the frequency dependence of the shielding effect of the epoxy- (PANI/ wollastonite-colemanite) composites in the frequency range of 0-8 GHz. Among the PANI/ wollastonite-colemanite composites, It is observed that epoxy-(wollastonite-colemanite (25-75 wt. %) /Aniline: 1/1) has more visible efficacy on microwave shielding effectiveness properties than other composites. The PANI/ wollastonite-colemanite compositions (wollastonite-colemanite (25-75 wt. %) /Aniline: 1/1) powders and epoxy indicated just one band at 6.26 GHz with -41.65 dB. In addition, it accomplished a shielding effect which is less than -10 dB in the frequency bands among 2.72 GHz and 3.95 GHz, 4.72 Ghz and 6.51 GHz, 6.65 GHz and 7.05 GHz, 7.59 GHz and 8 GHz (Fig3.a). Besides this composite material showed a shielding effect value less than -20 dB in the frequency band between 0 GHz and 0.61 GHz. When powder content is equal with aniline, the epoxy-(PANI / wollastonite-colemanite) compositions (wollastonite: colemanite (50-50 wt. %) /Aniline: 1/1) reach to -39.84 dB at 6.24 GHz (Fig3.b). Moreover, this composite material achieved a shielding effect of below – 10 dB in the frequency band between 0 GHz and 0.69 GHz, 1.14 GHz and 1.36 GHz, 5.55 GHz and 6.47 GHz, 7.77 GHz and 8 GHz, respectively and this composite achieved a shielding effectiveness less than -20 dB in the frequency bands among 6.09 GHz and 6.39 GHz. The epoxy-(PANI/ wollastonite-colemanite) compositions (wollastonite: colemanite (75-25 wt. %) /Aniline: 1/1) reach to a shielding effectiveness of -36.81 dB at 6.24 GHz (Fig3.c). Furthermore, it achieved a shielding effect less than – 10 dB in the frequency bands between 0 and 0.36 GHz, 3.48 GHz and 4.29 GHz, 5.09 GHz and 6.51 GHz, 6.65 GHz and 7.06 GHz, 7.34 GHz and 8 GHz, respectively. Besides, this composite acquired a shielding effect less than -20 dB in the frequency band between 5.73 GHz and 6.3 GHz.

Impedance matching was achieved through the use of polyaniline. PANI has effected the shielding effectiveness. Additionally, PANI and composite components play a great role in the efficacy of the electromagnetic shielding material. The efficacy of microwave shielding often be attached to adapting the irradiation impedance to the surface of the material. PANI raises the matching impedance of connections between the composite supplementals. Meanwhile, due to the strength of the conductive polymer PANI between composite components, sharp peaks of shielding effectiveness occur.

New epoxy-(PANI/wollastonite-colemanite) compositions were fabricated, polyaniline based wollastonite-colemanite composites have 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-wollastonite and PANI in this process. Researches indicate that the content of wollastonite: colemanite and PANI affect structure. Content of wollastonite-colemanite influenced permeability of material and add to the shielding effectiveness.

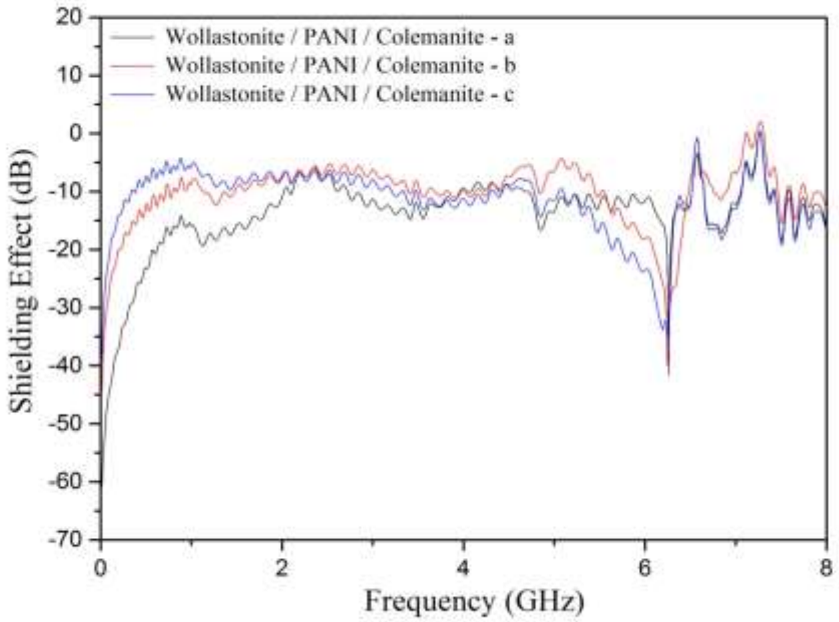

Figure 2. Microwave shielding effectiveness of the epoxy-(PANI / wollastonite-colemanite) composites: a) wollastonite- colemanite compositions (wollastonite-colemanite (25-75 wt. %) /Aniline: 1/1, b) wollastonite-colemanite compositions (wollastonite-colemanite (50-50 wt. %) /Aniline: 1/1 c) wollastonite-colemanite compositions (wollastonite-colemanite (75-25 wt. %) /Aniline: 1/1.

4. Conclusions and Recommendations

Wollastonite-colemanite powders (at 25-75 wt. %, 50-50 wt. % and 75-25 wt. %, respectively) were produced by using mixed oxide technique and PANI/ (wollastonite-colemanite) composites were obtained for the first time in research, to the best of our information.

The microwave shielding effectiveness characteristic was fabricated as 6.26 GHz and 1.5 mm in thickness with the minimal SE of -41.65 dB by the epoxy-(PANI/wollastonite-colemanite) compositions (wollastonite-colemanite (50-50 wt. %) /Aniline: 1/1).

Microwave shielding properties can be modulated easily by controlling the PANI content and the effect of wollastonite-colemanite content on the samples for the necessary frequency bands. Due to the simple and low cost preparation techniques and better shielding effectiveness performance, the PANI / wollastonite-colemanite composites have a encouraging potential as microwave shielding efficiency. Content of wollastonite-colemanite and Polyaniline were used to enhance the microwave shielding effectiveness.

Microwave shielding characteristics of PANI based wollastonite-colemanite composites indicate a strong variability with high concentrations of wollastonite-colemanite content. The best shielding effect value is obtained from epoxy-(wollastonite-colemanite (25-75 wt. %) /Aniline: 1/1) composition at the value of less than -10 dB and between 0 GHz and 2.01 GHz, 4.72 GHz and 6.51 GHz. This composite achieved a shielding effect less than -20 dB in the frequency band between 0 GHz and 0.61 GHz. The second shielding effect performances are obtained from epoxy- (wollastonite-colemanite (75-25 wt. %) /Aniline: 1/1) and epoxy-(wollastonite-colemanite (75-25 wt. %) /Aniline: 1/1) compositions at the value of less than -10 dB and between 0 GHz and 0.69 GHz, 5.55 GHz and 6.47 GHz. This composite reached a shielding effect less than -20 dB in the frequency band between 5.73 GHz and 6.3 GHz.

The polyaniline content exhibits an respectable role in variation of the shielding effectiveness. The microwave shielding effect features of the PANI / wollastonite-colemanite compositions can be researched for a wider range of concentration in this analysis. As PANI content is equal to the total powder amount, and the wollastonite-colemanite powder amounts change among themselves, the sheilding effect rised. Matching of impedance has been acquired by using PANI. PANI effect has been increased the shielding effectiveness. This study is evaluated compound of wollastonite and colemanite as a possible filling or replacement of polianilin for shielding materials. Literature on this topic is very limited. This style of composite has been produced for the first time for this purpose.

PANI based wollastonite-colemanite can be regarded as a candidate for microwave shielding effectiveness in a wideband frequency. The formation of wollastonite-colemanite compound content is a further field of research area. Wollastonite-colemanite and PANI content are being used in order to enhance the microwave shielding effect characteristics. The microwave shielding effectiveness and microwave reflection loss of PANI based wollastonite-colemanite in radar frequency and higher frequency ranges may be investigated.

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