

Categoriy: STEM (Science, Technology, Engineering and Mathematics)

ORIGINAL

Dielectric Properties of Three Perovskite Oxides Included in Three Types of Polymer Composites

Propiedades dieléctricas de tres óxidos de perovskita incluidos en tres tipos de compuestos poliméricos

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Cite as: Faeq LS, Farid SBH, Hashim FA. Dielectric Properties of Three Perovskite Oxides Included in Three Types of Polymer Composites. Salud, Ciencia y Tecnología - Serie de Conferencias. 2024; 3:866. https://doi.org/10.56294/sctconf2024866

Submitted: 04-02-2024

Revised: 17-04-2024

Accepted: 10-06-2024

Published: 11-06-2024

Editor: Dr. William Castillo-González 回

Note: paper presented at the 3rd Annual International Conference on Information & Sciences (AICIS'23).

ABSTRACT

Microwave dielectric double perovskite /polymer composites are materials that combine the flexibility of polymer matrices like epoxy, polyurethane, or silicone rubber with unique properties of double perovskite ceramics, such as Ba_2ZnWO_6 , Ba_2MgWO_6 , and $Bi_2MO_{0,5}W_{0,5}O_6$. These complex oxide ceramics are synthesized using the solid-state reaction technique. The X-ray diffraction analysis (XRD) confirmed the formation of the double perovskite phase with well-defined crystal structures. Utilizing transmission/reflection measurements by a vector network analyzer (VNA) and double perovskite ceramics as filler materials in the polymer matrix, the impact of the complex oxide fillers on the composites' overall dielectric properties was fully measured. In particular, the dielectric properties of the resulting composites, specifically their dielectric loss and dielectric constant, were investigated within the wide frequency range of 4-8 GHz. Better dielectric characteristics, such as a high dielectric constant and a low loss factor, were demonstrated by Ba₂ZnWO₄, which made it a viable option for use in high-frequency circuits, microwave devices, energy storage components and communication technology. Furthermore, Competitive dielectric qualities were demonstrated by Ba₂MgWO₄ and Bi₂Mo_{0.5}W_{0.5}O₆. In addition, the results indicated improved dielectric characteristics, including dielectric constant and loss factor. The resulting composites not only have a lower cost and light weight than ceramics, but they also have a lot of potential uses in the microwave dielectric range. Furthermore, when compared to the other measured composites the Ba₂ZnWO₂/polymer composites demonstrated superior properties.

Keywords: Dielectric Constant; Loss Factor; Perovskite Ceramics; Composites; Communication Technology.

RESUMEN

Los compuestos de perovskita doble /polímero dieléctrico para microondas son materiales que combinan la flexibilidad de las matrices poliméricas como el epoxi, el poliuretano o el caucho de silicona con las propiedades únicas de las cerámicas de perovskita doble, como Ba₂ZnWO₆, Ba₂MgWO6 y Bi₂Mo_{0,5}W_{0,5}O₆,. Estas cerámicas de óxidos complejos se sintetizan mediante la técnica de reacción en estado sólido. El análisis de difracción de rayos X (XRD) confirmó la formación de la fase doble perovskita con estructuras cristalinas bien definidas. Utilizando medidas de transmisión/reflexión mediante un analizador vectorial de redes (VNA) y cerámicas de doble perovskita como materiales de relleno en la matriz polimérica, se midió completamente el impacto de los rellenos de óxido complejo en las propiedades dieléctricas globales de los materiales compuestos. En particular, se investigaron las propiedades dieléctrica, dentro del amplio rango de frecuencias de 4-8 GHz.

© 2024; Los autores. Este es un artículo en acceso abierto, distribuido bajo los términos de una licencia Creative Commons (https:// creativecommons.org/licenses/by/4.0) que permite el uso, distribución y reproducción en cualquier medio siempre que la obra original sea correctamente citada El Ba2ZnWO6 demostró mejores características dieléctricas, como una elevada constante dieléctrica y un bajo factor de pérdida, lo que lo convirtió en una opción viable para su uso en circuitos de alta frecuencia, dispositivos de microondas, componentes de almacenamiento de energía y tecnología de comunicaciones. Además, Ba₂MgWO6 y Bi₂Mo_{0.5}W_{0.5}O₆ demostraron cualidades dieléctricas competitivas. Además, los resultados indicaron una mejora de las características dieléctricas, incluidas la constante dieléctrica y el factor de pérdida. Los compuestos resultantes no sólo son más baratos y ligeros que los cerámicos, sino que también tienen muchas posibilidades de uso en el rango dieléctrico de las microondas. Además, en comparación con los otros materiales compuestos medidos, los materiales compuestos Ba₂ZnWO₆/polímero demostraron propiedades superiores.

Palabras clave: Constante Dieléctrica; Factor de Pérdida; Cerámicas Perovskita; Composites; Tecnología de Comunicaciones.

INTRODUCTION

Over the past decade, the landscape of wireless communication has undergone a significant transformation, shifting from a predominantly military-driven domain to a commercially viable market with a focus on consumers. This evolution has prompted electronic industries to concentrate on achieving cost-effective product miniaturization, introducing new challenges and requirements.^(1,2,3,4,5,6,7,8) Amidst this backdrop, the expansion of telecommunication systems and high-speed digital devices has spurred scientific exploration into novel materials capable of delivering enhanced performance.

Despite the commendable thermal and dielectric properties found in many ceramic materials, their inherent brittleness constrains the range of applications they can serve. Conversely, certain polymers, crafted at low temperatures, exhibit remarkable mechanical properties, suggesting their potential as microwave substrates. Nevertheless, their utility is hampered by low thermal conductivity, low relative permittivity, and significant thermal expansion.^(1,2,3)

Addressing these challenges, the concept of composite materials involves merging the distinctive properties of ceramics with polymers to create ceramic-polymer composites tailored for electrical applications. Notably, combining polymer matrices with double perovskite ceramics provides an avenue to fine-tune the dielectric properties, catering to specific application requirements. Through careful selection of filler material and volume percentage, control over the dielectric constant and loss factor is achieved, optimizing efficiency in high-frequency applications. These composites exhibit versatility across various polymer matrices, including silicone rubber, epoxy, and polyurethane, owing to their exceptional dielectric characteristics.

Moreover, the literature review on perovskites underscores a significant disparity in reported dielectric parameter values for this ceramic material. Double perovskites Ba₂MeWO₆ (Me=Mg, Ni, Zn) were synthesized through conventional solid-state reaction over a broad temperature range. Single-phase ceramics were achieved at lower temperatures, around 1200°C, while higher temperatures resulted in the presence of minor second phases. Dielectric measurements revealed a positive temperature coefficient of permittivity for Ba,ZnWO, and Ba,MgWO, suggesting the impact of second polar modes involving B-site ion vibrations on their low-frequency dielectric properties.⁽⁹⁾ Double perovskite compounds (DPCs) have garnered attention for their versatility in electronics and spintronic. Employing density functional theory (DFT), we explored the electronic, magnetic, and optical properties of DPC $La_2BB'O_6$, where B = Cr, Sc, and V, and B' = Co, Ni. Electronic band gaps suggest these compounds exhibit half-metallic (HF) semiconducting behavior in the spin-up channel and metallic behavior in the spin-down channel. Magnetic properties indicate they are ferromagnetic, classifying all DPCs as half-metallic ferromagnetic (HM-FM). Notably, La, CrCoO, displays exceptional electronic and optical properties, making it suitable for optoelectronic/spintronic devices.⁽¹⁰⁾ 2 BWO₄ (A= Sr, Ba; B= Co, Ni, Zn) microwave dielectric ceramics were synthesized through conventional solid-state ceramic methods. X-ray diffraction (XRD) analysis confirmed a cubic perovskite structure for all compounds. Dielectric properties were measured in the 6.5 to 8.5 GHz frequency range using resonance methods, revealing dielectric constants within 18 to 30, high-quality factor (Q× f value up to 56 000 GHz), and negative temperature coefficients of resonant frequencies ranging from -72,9 to -31,1 ppm/C. The dielectric constant increased with B 2+ ionic polarizability, and enhanced quality factor resulted from increased B-site ordering. The relationship between temperature coefficient of dielectric constant and tolerance factor was consistent compared to other perovskites.⁽¹¹⁾ The study explores the structure, sintering behavior, and microwave dielectric properties of $A1-3x/2Lax(Mg_{4,0}W_{1,0})$ O, (A = Ba, Sr, Ca; $0,0 \le x \le 0.05$) ceramics. Utilizing XRD, Raman, SEM, and a network analyzer, dielectric constants within the range of 19-20 were observed for all compounds. Q × f values exhibited significant variation based on bulk density, B-site ordering, and the presence of a second phase. The absolute value of τf decreased in the order of BLMW, SLMW, and CLMW series. Exceptional combination microwave dielectric properties were

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achieved for the BLMW sample with x = 0,04, sintered at 1450 °C/2 h: $\varepsilon r \sim 20$, Q × f = 87,680 GHz, and $\tau f = -1,2$ ppm/°C.⁽¹²⁾ The microwave dielectric ceramics Ba2Ca1-xSrxWO6 (x=0-1) were synthesized using the twostep solid-state reaction method. The dielectric constant (εr) remained nearly unchanged, the quality factor (Q × f) consistently decreased, and the temperature coefficient of resonant frequency (τf) changed sign with increasing x. A favorable combination of microwave dielectric properties was achieved for Ba₂Ca_{0,975}Sr_{0,025}WO₆ sintered at 1250°C: $\varepsilon r=23,9$, Q × f=80,200 GHz and $\tau f=+18$ ppm/°C.⁽¹³⁾ However, prior studies on perovskite ceramics as filler materials in polymer matrices, particularly with epoxy, polyurethane, and silicone rubber, are limited. The present article aims to fabricate cost-effective microwave materials with investigated properties. Specifically, epoxy, polyurethane and silicone rubber/ Ba₂MgWO₆, Ba₂ZnWO₆, and Bi₂Mo_{0,5}W_{0,5}O₆ composites were considered in the current study.

Experimental Works

In this work, polymer-ceramic composite materials were prepared by a simple hand-mixing technique. More precisely, the utilized polymers were (epoxy, polyurethane, and silicone rubber). Each polymer is blended with 5 % of Ba_2MgWO_6 , Ba_2ZnWO_6 , and $Bi_2Mo_{0.5}W_{0.5}O_6$. The synthesis of Ba_2MgWO_6 polycrystalline powder, high-purity BaCO., MgO, and WO, were combined in a stoichiometric ratio suitable for the desired Ba.MgWO. composition. Particularly, the average particle diameters of oxides as measured by the AFM technique were 44,04 nm, 37,27 nm, and 44,07 nm, respectively. The oxide mixture was mixed in an electrical blender for six hours. Subsequently, the mixture was calcined at 1200°C for three hours and slowly cooled down to room temperature to avoid thermal stress. After calcination, the powders were milled and mixed for six hours. Then, the obtained powders were characterized using XRD to confirm their composition. Likewise, Ba₂ZnWO₂ was prepared following a similar procedure as that of Ba₂MgWO₆, using the ZnO powder with an average diameter of 47,78 nm. Moreover, the synthesis of Bi₂Mo_{0.5}W_{0.5}O₆ was done using high-purity Bi₂O₃, WO₃, and MoO₃ powders with average diameters of 33,37 nm, 44,07 nm, and 34,35 nm, respectively. In particular, the oxides were weighed in appropriate stoichiometric ratios. After that, the oxide mixture was milled and mixed using the electrical blender for two hours. The resulting mixture was then calcined at 650°C for two hours, followed by re-milling for two hours. The XRD analysis confirmed the compositions of the obtained powders. To assess the phase compositions of raw materials and complex oxides, the XRD analysis with Cu ka radiation was used. Furthermore, the dielectric properties of the polymer-ceramic composites were analyzed using a Transmission/Reflection method in the frequency range of 4-8 GHz utilizing a network analyzer. Additionally, the Nicholson-Ross-Weir (NRW) method was utilized to convert the scattering parameters (S_{11} and S_{21}) into dielectric properties. ^(14,15,16) To the best of the authors' knowledge, there has been no examination or investigation into the dielectric properties of $Bi_2Mo_{0.5}W_{0.5}O_{6.5}$

RESULTS AND DISCUSSION

Figure 1 shows the XRD pattern of Ba_2MgWO_6 , Ba_2ZnWO_6 , and $Bi_2Mo_{0,5}W_{0,5}O_6$. These diffractgrams show that the prepared compounds were crystallized at proper phases, as mentioned in the PDF cards. Table 1 shows the unit cell parameters and the corresponding powder diffraction PDF file.





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Figure 1. (a) The X-ray diffraction patterns of Ba_2MgWO_6 ceramic, (b) The XRD pattern of Ba_2ZnWO_6 ceramic (c) The XRD pattern of $Bi_2Mo_{0.5}W_{0.5}O_6$

Table 1. The unit cell parameters and the corresponding powder diffraction PDF file of the complex oxides				
Complex oxides	Space group	Unit cell parameters	PDF no.	
Ba ₂ MgWO ₆	space group Fm- 3m (225)	<i>a=b=c</i> =8,11800 A° α =90°, β =90°, γ =90°	96-153-7303	
Ba ₂ ZnWO ₆	space group Fm- 3m (225)	<i>a=b=c</i> =8,11800 A° <i>α</i> =90°, <i>β</i> =90°, <i>γ</i> =90°	96-152-7205	
${\rm Bi_2Mo_{0,5}W_{0,5}O_6}$	P c a 21(29)	a= 5,43726 A° ,b= 16,43018 A° and c= 5,45842 A° for Bi_2WO_6 and a= 5,48220 A°, b= 16,19860 A° and c= 5,50910 A° for Bi_2MOO_6	96-901-1352 96-901-1800	

Moreover, figure 2 shows the dielectric constant of the blank polymers. On the other hand, figure 3 compares the dielectric constant of 5 % Ba₂MgWO₂/polymer composites with that of 5 % Ba₂ZnWO₂/polymer composites. In this regard, the dielectric constant of Ba₂ZnWO₆/polymer composites exhibits an increase at the frequency range of 7-7,5 GHz In particular, Zn is known to exhibit higher polarizability than that of Mg. In this context, polarizability refers to the ease with which the electron cloud around an atom can be distorted by an applied electric field. Since Zn has more electrons and a larger atomic radius than those of Mg, it tends to have higher polarizability and can undergo greater distortion. This increased ability to polarize in Ba, ZnWO, contributes to a higher dielectric constant in the reinforced polymer. Figure 3a illustrates the composite's Ba₂MgWO₂ particles displaying a comparatively high dielectric constant of 46,9 at 5,56GHz, which might be a result of the material's inherent characteristics, such as its elemental makeup and crystal structure, which, under the effect of the applied electromagnetic field, encourage stronger polarization. Moreover, the relaxation time of the material and its interaction with the epoxy matrix are two potential causes of the reduction in the dielectric constant at 6,96 GHz with a value of 20,4. In particular, the material's response to the electric field might be less effective at this frequency, resulting in a lower dielectric constant. In figure 3b, Ba₂ZnWO₆ has a greater dielectric constant with a value of 83,3 at 7,1GHz over that of Ba,MgWO, which could be because of the variations in its elemental make-up, electricity characteristics, or crystal structure. To this end, Ba,ZnWO, is a good choice for applications that call for high dielectric constants at this particular frequency since the composite's dielectric constant at the peak frequency shows that it possesses strong polarization properties. Figure 3c and d illustrate the considerable difference in the dielectric constant between the polyurethane/Ba,MgWO, and the polyurethane/Ba, ZnWO, composites at various frequencies. Specifically, at 4,48 GHz, the Ba, MgWO, particles with the polyurethane matrix composite exhibit a relatively low dielectric constant value of 12,75. In contrast, the polyurethane/Ba,ZnWO, composite demonstrates a significantly higher dielectric constant value of 86,54 at 7,12 GHz. In figure 3e, the silicone rubber/Ba,MgWO, composite exhibits a relatively high dielectric constant at 5,58 GHz with a value of 50,4. On the other hand, the silicone rubber/Ba,ZnWO_c composite exhibits an even higher dielectric constant value of 57,3, as shown in figure 3f. Specifically, Ba, ZnWO, appears to have strong polarization at 7,1 GHz, resulting in a high dielectric constant compared to that of Ba,MgWO,.



Figure 2. The dielectric constant calculated at a frequency range of 4-8 GHz for (a) blank epoxy (b) blank polyurethane (c) blank silicone rubber





Figure 3. The dielectric constant calculated at a frequency range of 4-8 GHz for (A) 5 % vf Ba₂MgWO₆ / epoxy composite
(B) 5 %vf Ba₂ZnWO₆ / epoxy composite (C) 5 % vf Ba₂MgWO₆ / polyurethane composite (D) 5 % vf Ba₂ZnWO₆ / polyurethane composite (E) 5 % vf Ba₂MgWO₆ / silicone rubber composite (F) 5 % vf Ba₂ZnWO₆ / silicone rubber composite

As shown in figure 4, $Bi_2Mo_{0,5}W_{0,5}O_6$ is a complex oxide and has high dielectric constants. Particularly, the presence of molybdenum and tungsten in the compound contributes to its enhanced polarizability, and thus, its higher dielectric constant. While Ba_2MgWO_6 (BMW) contains magnesium as one of its cations, and while it is a divalent cation, it may not provide the same level of polarizability as that of the combination of molybdenum and tungsten in $Bi_2Mo_{0,5}W_{0,5}O_6$. Consequently, BMW tends to have a lower dielectric constant compared to that of $Bi_2Mo_{0,5}W_{0,5}O_6$. Figure 4d shows the dielectric constant of $Bi_2Mo_{0,5}W_{0,5}O_6$ /polyurethane composite, which is 48 at 7,4 GHz. At 6,34 GHz, the dielectric constant is 102 for $Bi_2Mo_{0,5}W_{0,5}O_6$ /silicone rubber composite, indicating a quite high dielectric constant for this frequency. At 7,54 GHz, the dielectric constant is 18,5.

From the above discussion, this study implies that the frequency, the type of the polymer matrix, and the complex oxide have an important effect on the dielectric characteristics of the materials. Consequently, when constructing electrical components or devices, the frequency range and the composite material selection are crucial for ensuring the best performance for a wide range of applications.





Figure 4. The dielectric constant calculated at a frequency range of 4-8 GHz for (A) 5 % vf $Bi_2Mo_{0,5}W_{0,5}O_6$ / epoxy composite (B) 5 % vf Ba_2MgWO_6 / epoxy composite (C) 5 % vf Ba_2MgWO_6 / polyurethane composite (C) 5 % vf $Bi_2Mo_{0,5}W_{0,5}O_6$ / polyurethane composite (E) 5 % vf Ba_2MgWO_6 / silicone rubber composite (F) 5 % vf $Bi_2Mo_{0,5}W_{0,5}O_6$ / silicone rubber composite

Figure 5 shows the loss tangent values of the blank polymers. In addition, figure 6 shows that the loss tangent values indicate the extent of energy dissipation in the respective materials. More precisely, a lower loss tangent value suggests that the material exhibits lower energy dissipation and is more efficient in terms of dielectric performance. The comparison between the losses tangent of these composites suggests that the Ba_2ZnWO_6 /polymer composites have a lower loss tangent than that of the Ba_2MgWO_6 /polymer composites, which implies that the Ba_2ZnWO_6 -based composites exhibit reduced energy dissipation and potentially better dielectric properties compared to those of the Ba_2MgWO_6 - based composites.



Figure 5. The loss tangent calculated at a frequency range of 4-8 GHz for (A) blank epoxy (B) blank polyurethane (C) blank silicone rubber



Figure 6. The loss tangent calculated at a frequency range of 4-8 GHz for (A) 5 % vfBa₂MgWO₆ / epoxy composite (B) 5 % vf Ba₂ZnWO₆ / epoxy composite (C) 5 % vf Ba₂MgWO₆ / polyurethane composite (D) 5 % vf Ba₂ZnWO₆ / polyurethane composite (E) 5 % vf Ba₂MgWO₆ / silicone rubber composite (F) 5 % vf Ba₂ZnWO₆ / silicone rubber composite

Figure 7 demonstrates that as compared to Ba_2MgWO_6 , $Bi_2Mo_{0.5}W_{0.5}O_6$ often has a lower loss factor. With respect to flaws and energy dissipation sources, $Bi_2Mo_{0.5}W_{0.5}O_6$ in particular might have a better ordered crystal structure. The fact that the loss factor is smaller suggests that $Bi_2Mo_{0.5}W_{0.5}O_6$ is more effective in storing and minimizing energy losses while releasing electrical energy. But BMW is a different story. Decent dielectric substance with little capabilities for energy dissipation; additionally, it might a loss factor that is somewhat more than $Bi_2Mo_{0.5}W_{0.5}O_6$. In the event, that the presence of magnesium in the crystal structure could be a factor in the higher energy dissipation. The results are concisely outlined in table 2.



Figure 7. The loss tangent calculated at a frequency range of 4-8 GHz for (A) 5 % vf Ba₂MgWO₆ / epoxy composite (B) 5 % vf Bi₂Mo_{0,5}W_{0,5}O₆ / epoxy composite (C) 5 % vf Ba₂MgWO₆ / polyurethane composite (D) 5 % vf Bi₂Mo_{0,5}W_{0,5}O₆ / polyurethane composite (E) 5 % vf Ba₂MgWO₆ / silicone rubber composite (F) 5 % vf Bi₂Mo_{0,5}W_{0,5}O₆ / silicone rubber composite

In comparison to the unfilled polymer counterparts as shown in figure 2, there is a substantial enhancement in the dielectric properties of the composites due to different things. Ceramics often have a high intrinsic dielectric constant because they are insulators with a large number of electric dipoles. When these ceramics are incorporated into a polymer matrix, they introduce additional polarizability to the composite. This leads to increased dielectric constant because polarization is a key factor in determining the dielectric behavior of materials.At the interface between ceramic particles and the polymer matrix, there can be localized electric fields due to differences in permittivity. This interfacial polarization effect can significantly contribute to an enhanced dielectric constant.The presence of two or more materials with significantly different dielectric constants in a composite can lead to the MWS effect (Maxwell-Wagner-Sillars). This effect arises from charge accumulation at interfaces and contributes to higher dielectric constants in composites.^(17,18)

Table 2. Summary of results					
Polymer-ceramic	Frequency (GHz) as a function of filler volume fraction (%)	Dielectric constant as a function of filler volume fraction (%)	Loss tangent as a function of filler volume fraction (%)		
	5 %	5 %	5 %		
Epoxy/ Ba ₂ MgWO ₆	5,56	46,9	0		
Polyurethane / Ba ₂ MgWO ₆	4,48	12,75	0		
Silicone Rubber/ Ba ₂ MgWO ₆	5,58	83,3	0		
Epoxy/ Ba ₂ ZnWO ₆	7,1	86,54	0		
Polyurethane / Ba ₂ ZnWO ₆	7,12	57,3	0		
Silicone Rubber/ Ba ₂ ZnWO ₆	7,1	8	0		
Epoxy/Bi ₂ Mo _{0,5} W _{0,5} O ₆	5,24 6,76 7,66 7 6,2 6,38 7,86	6,64 76,43 11,43 12,3 11,87 48,1 15,9	0,183 0,148 0,0574 -0,561 -1,48 -1,23 -9,92		
Polyurethane / $\mathrm{Bi_2Mo_{0,5}W_{0,5}O_6}$	7,4 6,4	102 18,5	1,99 -0,942		
Silicone Rubber / $Bi_2Mo_{0,5}W_{0,5}O_6$	6,34 7,54		0,0824 0,183		

CONCLUSIONS

In this work, a solid state reaction technique was effectively used to synthesize microwave dielectric ceramics, such as Ba_2MgWO_6 , Ba_2ZnWO_6 , and $Bi_2Mo_{0,5}W_{0,5}O_6$, meeting the standard PDF cards. To create composites, these ceramics were subsequently manually mixed into polymer matrices. These composites' microwave dielectric characteristics were assessed with a vector network analyzer, and the Nicholson-Ross Weir (NRW) method was used to determine their dielectric constant. Notably, the addition of ceramics Ba_2MgWO_6 , Ba_2ZnWO_6 , and $Bi_2Mo_{0,5}W_{0,5}O_6$ to the polymer matrices resulted in notable modifications to the dielectric characteristics in the (4-8) GHz C-band, including the dielectric constant and the loss tangent. In comparison to the Ba_2MgWO_6 /polymer composites showed a better dielectric constant and a lower loss factor at a frequency of 7-7,5 GHz and a filler volume percentage of 5%. At the same filler volume percentage and frequency, the $Bi_2Mo_{0,5}W_{0,5}O_6$ /polymer composites showed superior dielectric characteristics compared to the Ba_2MgWO_6 /polymer composites.

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FINANCING

The authors did not receive financing for the development of this research.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

AUTHORSHIP CONTRIBUTION

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