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EFFECT OF CERAMIC MICROSPHERES ON EPOXY POROUS SOUND-ABSORBING MATERIALS

VPLIV KERAMIČNIH MIKROKROGLIC NA POROZNE EPOKSIDNE MATERIALE ZA ABSORPCIJO ZVOKA

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In order to study the effect of ceramic microspheres on the sound absorption, sound insulation, abrasion resistance and tensile properties of the material, specimens with proportions of ceramic microsphere components of (0, 20 and 40) % were prepared for the experiments. The results show that an appropriate addition of ceramic microspheres can improve the pore structure of the material and enhance its sound absorption; however, excessive addition can block the pores and consequently degrade the material's performance. Comparing the sound absorption and sound insulation performance of three groups of samples, it was found that EM-0 and EM-20 exhibited better sound absorption performance. The sound insulation of EM-20 containing ceramic microsphere material increased from 20.64 dB to 22.7 dB compared to EM-0. Therefore, the addition of ceramic microspheres improved the sound insulation performance of the sound-absorption material. According to physical performance tests, the compressive strength of the EM-20 sample is 0.85 MPa, the tensile strength is 0.6 MPa, the wear is 0.335 g/cm^2, the average porosity is 71.5 %, and the water absorption is 45.9 %. Therefore, the density of the sample is relatively low, making it easy to transport. From this, it can be concluded that ceramic microspheres can optimize the overall performance of materials, and the most suitable amount of ceramic microspheres added is 20 %, achieving the best sound absorption effect.

Key words: ceramic microspheres, mechanical properties, organization analysis, porous sound-absorbing materials

Avtorji v članku opisujejo študijo vpliva dodatka keramičnih kroglic mikronske velikosti na absorpcijo zvoka, zvočno izolacijo, odpornost proti abraziji in mehanske lastnosti izbranega epoksidnega materiala. Za preizkuse so izbrali 20 % in 40 % dodatek mikronskih kroglic. Rezultati raziskav so pokazali, da dodatek mikronskih kroglic lahko izboljša strukturo por in izboljša absorpcijo zvoka. Toda prevelik dodatek kroglic blokira pore in obratno poslabša lastnosti materiala. Medsebojna primerjava absorpcije zvoka in zvočne izolacije treh skupin vzorcev je pokazala, da imata vzorca EM-0 in EM-20 boljšo absorpcijo zvoka. Zvočna izolacija vzorca EM-20 z 20 % dodatkom kroglic se je povečala z 20,64 dB na 22,7 dB v primerjavi z vzorcem brez dodatka kroglic EM-0. Tako avtorji zaključujejo, da dodatek keramičnih mikrokroglic izboljša zvočno izolacijo materiala za absorpcijo zvoka. Rezultati mehanskih preiskav so pokazali, da je tlačna trdnost vzorca EM-20 enaka 0,85 MPa, natezna trdnost 0,60 MPa, njegova abrazijska obraba 0,335 g/cm^2, povprečna poroznost 71,5% in absorpcija vlage 45,9%. Material ima relativno majhno gostoto in ga je zato enostavno transportirati. Rezultati raziskave so torej pokazli, da je z dodatkom keramičnih kroglic mikronske velikosti možno optimizirati lastnosti materialov in da z 20 %-nim dodatkom dosežemo najboljši učinek absorpcije zvoka.

Ključne besede: keramične kroglice mikronske velikosti, mehanske lastnosti, organizacijska analiza, porozni materiali za absorpcijo zvoka

1 INTRODUCTION

With the development of science and technology, noise pollution has become increasingly severe, affecting people's physical health. This noise pollution can cause not only various diseases such as heart disease, learning disabilities, and tinnitus but may also shorten lifespan.[1–3] Therefore, reducing noise pollution is crucial for protecting people's health. Besides controlling noise sources, measures to manage noise pollution include the application of sound-absorbing materials, such as resonant sound-absorbing materials and porous sound-absorbing materials.[2–6] Among them, porous sound-absorbing materials have become an ideal choice for noise control in fields such as construction and transportation due to their special properties like low cost, ease of molding, and light weight.[7–9]

Zhang et al.[10] added ceramic microspheres to a blend of polypropylene (PP) and polystyrene (PS), finding that the microspheres caused a significant reduction in the size of the PS phase and made its distribution more uniform. Tong et al.[11] found in their study that when ceramic microspheres were added as fillers to an immiscible polydimethylsiloxane (PDMS)/polyisobutylene (PIB) blend, the aggregation behavior of these nanoparticles was not only affected by their surface properties but also closely related to their concentration. Yang et al.[12] utilized ethylene-propylene-diene monomer (EPDM) and ceramic microsphere particles to prepare modified polypropylene plastics via one-step and two-step methods. They discovered that when a filler network structure formed, the mechanical properties of the polypropylene

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plastic were significantly enhanced. These findings provide new perspectives for optimizing ceramic microsphere porous sound-absorbing materials, aiming to find an ideal solution balancing mechanical properties and processability.

However, current porous sound-absorbing materials still face the challenge of insufficient mechanical properties, making them prone to breakage or wear during practical use. To address this, this study prepared five specimens with different ceramic microsphere compositions. The effects of the ceramic microsphere addition on sound absorption and insulation properties were evaluated by testing the samples. To investigate the intrinsic structural characteristics of the materials, Fourier transform infrared (FTIR) spectroscopy was used to analyze the chemical composition. The material structure was further understood by measuring water absorption, and calculating density and porosity. Tensile, compressive, and abrasion resistance tests were conducted to compare the performance of each group of specimens. These comprehensive experiments aimed to thoroughly analyze how ceramic microspheres alter the properties of epoxy-resin porous materials, providing a scientific basis for optimizing material design and practical applications.

2 EXPERIMENTAL MATERIALS AND METHODS

2.1 Experimental materials and equipment

This experiment used E-44 epoxy resin as the base material, ceramic microspheres as the filler, sodium dodecyl sulfate (SDS) as the foaming agent, and modified silicone resin polyether microemulsion as the foam stabilizer to prepare epoxy-resin porous sound-absorbing materials. The selection and proportion of these components significantly impact the sound absorption, sound insulation performance, and microstructure of the epoxy porous materials. The experimental equipment included an FS-400D high-speed disperser for stirring and mixing the components; a YLY-XH-100B (V3) ultrapure water system to reduce impurities during sample preparation; an S-4800 scanning electron microscope (SEM) to observe the micro-morphology of the material structure; an ASR-5611 abrasion tester to test the abrasion resistance and obtain the wear amount; an LD26.105 electronic universal testing machine to measure tensile and compressive properties; and an SWTM01-X sound absorption coefficient tester using the standing wave tube method to measure the sound absorption coefficient of the epoxy porous material.

2.2 Preparation of ceramic microsphere-epoxy resin porous sound-absorbing materials

To ensure the effectiveness of the foaming agent, an equal proportion of foam stabilizer was also added. From **Figure 1**, it can be seen that in the samples with ceramic microsphere composition ratios of (0, 10, 20, 30 and 40) % (named EM-0, EM-10, EM-20, EM-30, and EM-40, respectively), the molding effect of EM-0, EM-20, and EM-40 is significantly better than that of EM-10 and EM-30. Even after 48 hours, EM-10 and EM-30 still failed to reach the expected curing state, exhibiting numerous cracks on the surface; some fragments and powder even fell off, making specific testing impossible. This might have been due to an excessively low or high ratio of epoxy resin to curing agent, causing the curing reaction to be too slow or not initiating at all. In the EM-10 and EM-30 specimens, the presence of microspheres may have altered the viscosity and reactivity of the resin system, making it harder for the curing agent to distribute evenly, thereby affecting the curing process. In contrast, the EM-0, EM-20, and EM-40 specimens all reached a good curing state after 48 hours, with a smooth and flat surface, and no obvious cracks or fragments falling off. Therefore, only specimens with ceramic microsphere proportions of (0, 20 and 40) % were used in subsequent experiments, named EM-0, EM-20, and EM-40, respectively.

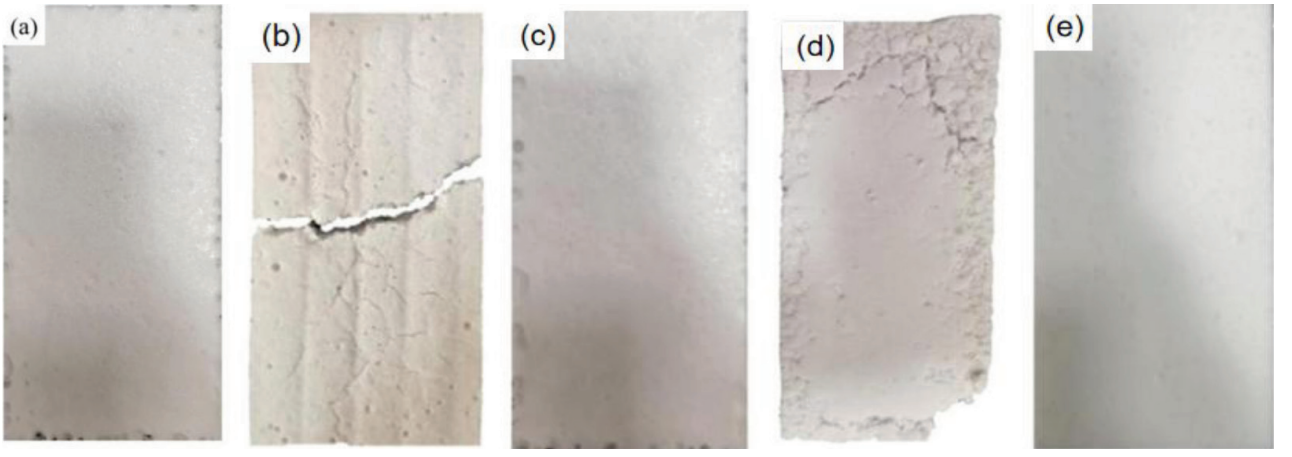

Figure 1: Samples with 0, 10, 20, 30 and 40 % of ceramic microspheres: (a) EM-0, (b) EM-10, (c) EM-20, (d) EM-30, (e) EM-40

3 RESULTS AND ANALYSIS

3.1 Effect of ceramic microspheres on microstructure and sound-absorption properties

(1) Effect of ceramic microspheres on sound-absorption performance

Figure 2 shows the sound-absorption coefficients of the materials with three different ceramic microsphere contents. From **Figure 2**, it can be seen that EM-20 has better sound-absorption performance, with an average sound-absorption coefficient of 0.82. EM-0 has relatively poorer sound absorption, with an average coefficient of 0.71. EM-40 exhibits the poorest overall sound-absorption performance, with an average coefficient of 0.52. By comparing the three curves, it can be concluded that the addition of ceramic microspheres can improve the sound-absorption performance of the material, but an excessive ceramic microsphere content reduces it. This is mainly because too high a content of ceramic microspheres blocks the internal pores of the material, reducing sound dissipation within the material and thereby lowering its sound-absorption performance.

As seen in **Figure 3**, when the test frequency is adjusted between 1000–2000 Hz, it is evident that the sound-absorption coefficient of EM-20 in this frequency range is higher than those of EM-40 and EM-0. Comparing the three absorption curves shows that an appropriate amount of ceramic microspheres enhances the sound-absorption performance of the material in the frequency range of 1000–2000 Hz, i.e., it improves the mid-frequency sound-absorption performance. Since the frequency of daily life noise is generally between 100–2000 Hz, this sound-absorbing material can significantly reduce the impact of noise on people's daily lives.

Figures 2 and **3** show that among the three sound-absorbing materials – EM-0, EM-20, and EM-40 – EM-20 exhibits the best sound-absorption performance, while EM-40 exhibits the poorest performance. Therefore, in the subsequent experiments, performance tests focused mainly on EM-0 and EM-20 to study the effect of ceramic microspheres on the properties of the sound-absorbing material.

(2) Effect of ceramic microspheres on sound-insulation performance

Tables 1 and **2** show the sound-insulation performance of two specimens of the same thickness at different frequencies. It can be seen that both EM-20 and EM-0 exhibit better sound insulation at 250 Hz. The sound-insulation performance decreases slightly as the frequency increases. At 2000 Hz, the average sound-insulation level of EM-20 is 21.6 dB, while that of EM-0 is 19.1 dB. Across the entire frequency band (200–2000 Hz), the average sound-insulation level of EM-20 is 22.7 dB, compared to 20.64 dB for EM-0. Therefore, the addition of ceramic microspheres improves the sound-insulation performance of the sound-absorbing material.

Table 1: Sound-insulation performance of EM-20 samples at different frequencies (dB)

Sample	250 Hz	500 Hz	1000 Hz	2000 Hz
1	24	21.1	21.7	20.8
2	26.5	21.3	22.4	21.6
3	24.3	22.7	21.6	21.6
4	27.8	22.4	21.2	22.3

Table 2: Sound-insulation performance of EM-0 samples at different frequencies (dB)

Sample	250 Hz	500 Hz	1000 Hz	2000 Hz
1	20	21.8	20.3	18.8
2	21.2	21.6	19.6	19.6
3	22.1	21.5	20.4	18.7
4	23.4	21.7	20.2	19.3

The improvement in sound-insulation performance shows that under the same conditions, sound-absorbing

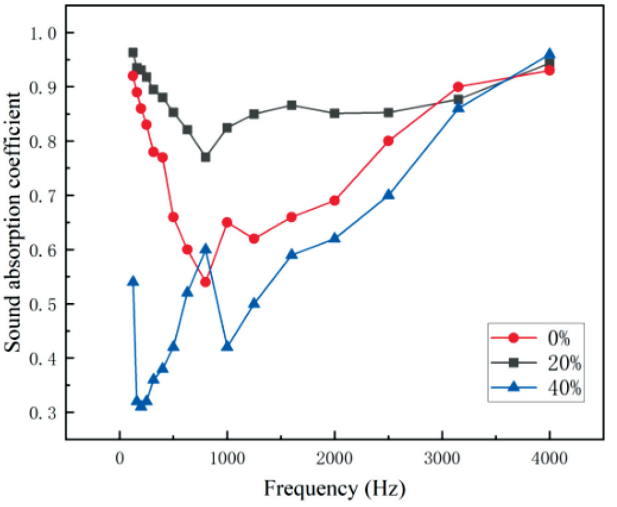

Figure 2: Sound-absorption coefficients of three materials with a ceramic microsphere content

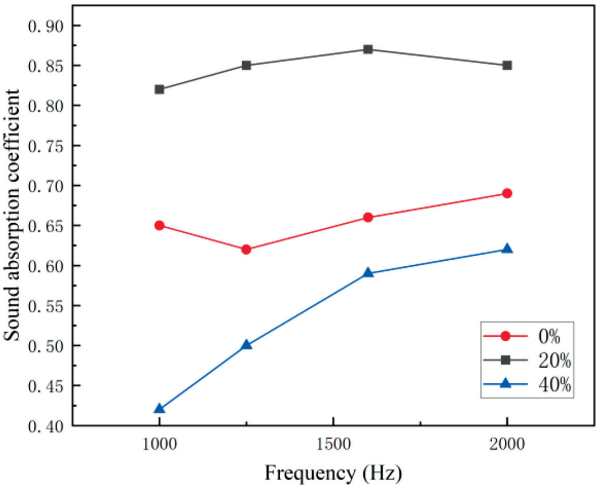

Figure 3: Sound-absorption coefficients of three materials with a ceramic microsphere content at 1000–2000 Hz

materials containing ceramic microspheres can more effectively reduce sound transmission, thereby providing a superior acoustic environment. This is particularly important in situations requiring high sound insulation, such as recording studios, concert halls, and conference rooms. Furthermore, although the sound-insulation performance of both types of specimens decreased in the high-frequency range (e.g., 2000 Hz), EM-20 still exhibited better performance. This further confirms the important role of ceramic microspheres in improving the sound-insulation performance of sound-absorbing materials.

(3) Effect of ceramic microspheres on the microstructure of sound-absorbing materials

Both EM-0 and EM-20 materials prepared in this experiment contain large-aperture pores, facilitating an easy entry of sound waves into the material's interior. This open-pore structure is one of the key features of sound-absorbing materials, as it allows sound waves to propagate and be impeded within the material, converting sound energy into thermal energy or another form of energy.

Secondly, the internal structure of the material shown in **Figure 4** reveals the presence of pores and channels of various sizes. This multi-level pore structure further enhances the sound-absorption performance because it can effectively scatter and impede sound waves at different frequencies. This means the material can absorb sound waves of different frequencies, thereby improving its overall sound absorption.

Finally, the difference in the inner walls of the pores observed via SEM is key to understanding the performance difference between EM-0 and EM-20. As shown in **Figure 5**, the inner walls of the pores in EM-20 are filled with ceramic microspheres of varying sizes, increasing the roughness of the pore walls. This rough inner wall surface can scatter sound waves more effectively, increasing the interaction between sound waves and the material's interior, thereby further improving sound absorption[13]. In contrast, the inner walls of the pores in EM-0 may be smoother, and their scattering effect on sound waves may be less effective than that of EM-20.

In summary, the differences in structure and performance between the EM-0 and EM-20 materials are mainly reflected in the roughness of the pore inner walls

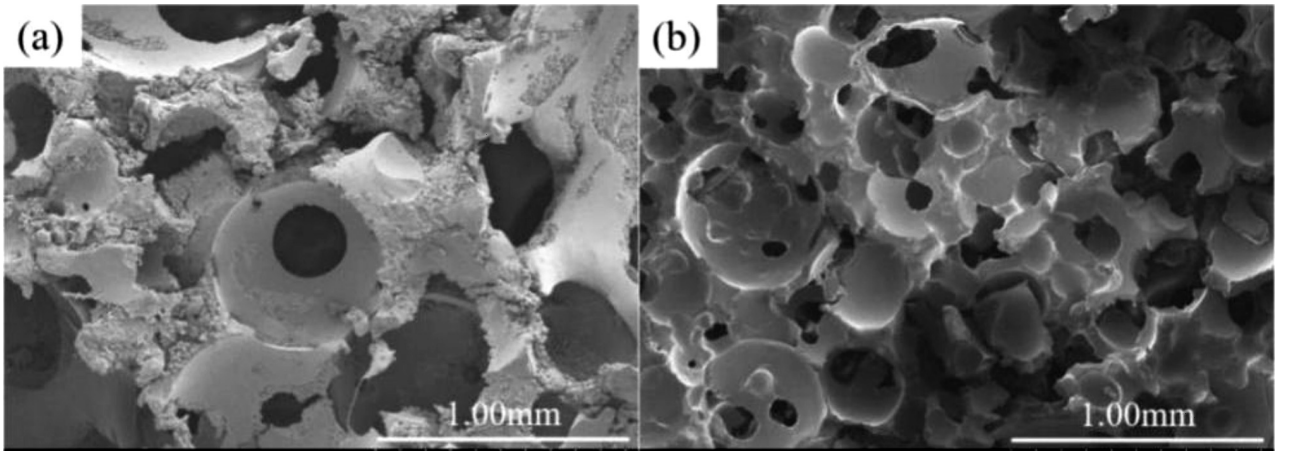

Figure 4: SEM comparison of two samples: a) EM-0, b) EM-20

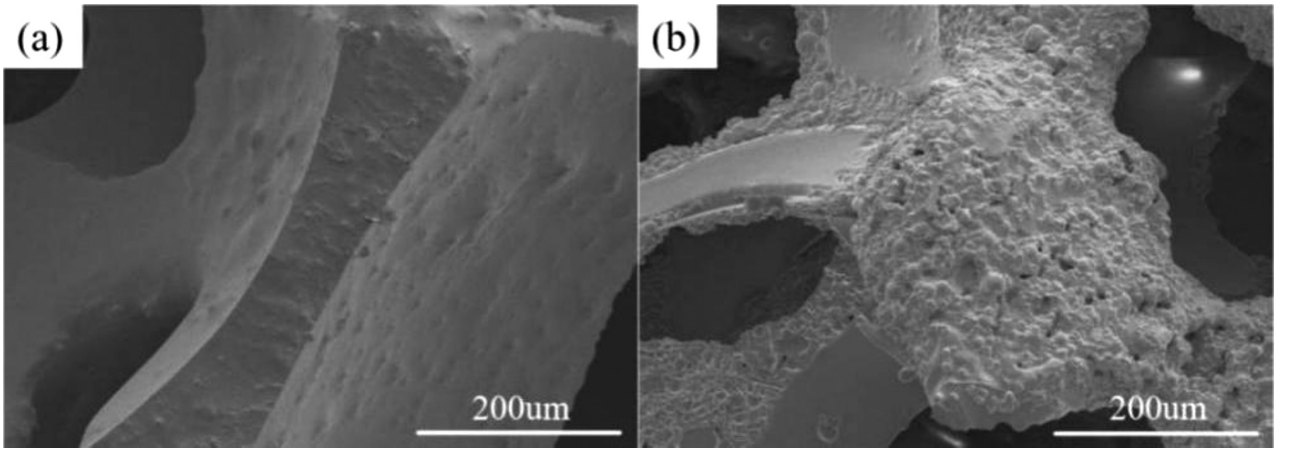

Figure 5: SEM comparison of the inner walls of the pores of the two samples: a) EM-0, b) EM-20

and the multi-level pore structure. These differences collectively influence the material's sound absorption, resulting in EM-20 exhibiting superior performance.

Composition analysis of the ceramic microspheres via SEM-EDS provides information on the elemental composition and relative content of the microsphere surface. The analysis revealed that oxygen (O), silicon (Si), and aluminum (Al) were the three most abundant elements, reaching (51.50, 25.67 and 9.22) %, respectively. These data provide an important basis for understanding the chemical composition and potential properties of the ceramic microspheres. **Figure 6** shows a schematic diagram of ceramic-microsphere test point selection, and **Figure 7** shows the SEM-EDS spectrum of the ceramic microspheres.

The high content of oxygen is common in ceramic materials, as ceramics are typically made from oxides or other oxygen-containing compounds. The high oxygen content suggests that the ceramic microspheres contain significant amounts of silicates or other oxides. Silicon is also a common element in ceramics, especially in silicate ceramics. Its content, second only to oxygen, further indicates that the microspheres are likely primarily composed of silicates. In ceramic materials, aluminum usually combines with other elements (such as oxygen and silicon) to form stable compounds like alumina or aluminosilicates. An addition of aluminum may affect the physical and chemical properties of ceramic microspheres, such as hardness, thermal stability, or electrical properties. To more accurately determine the composition of the ceramic microspheres, X-ray diffraction (XRD) analysis was also performed.

Table 3: Composition of ceramic microspheres

Element	*w*/%	*φ*/%	Chemical compound
C K	8.13	5.54	$CaCO_3$
O K	51.50	59.17	SiO_2
Na K	5.48	4.38	—
Al K	9.22	14.11	Al_2O_3
Si K	25.67	16.8	SiO_2

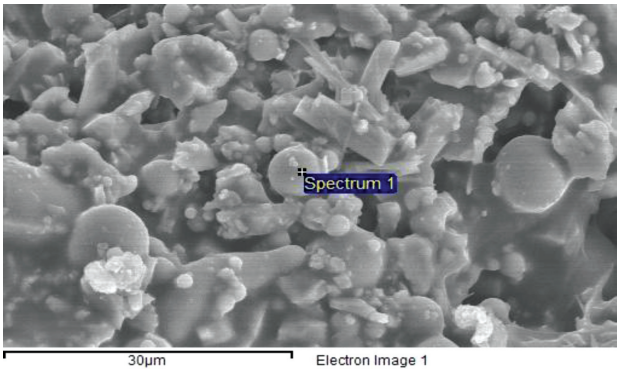

Figure 6: Schematic diagram of ceramic-microsphere test point selection

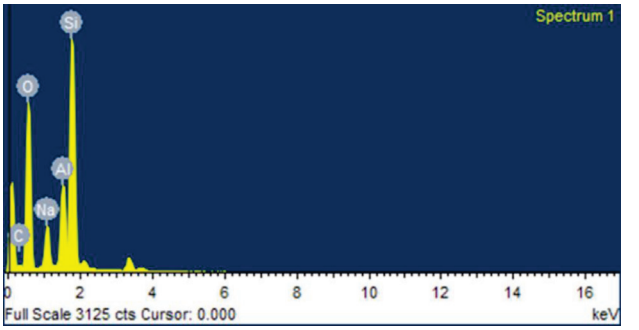

Figure 7: SEM-EDS spectrum of ceramic microspheres

Figure 8 shows the XRD pattern of the ceramic microspheres. Comparing the pattern with the International Centre for Diffraction Data (ICDD) standard cards revealed that it essentially matches the $CaAl_2Si_2$ PDF#72-2026 pattern. Combined with the SEM-EDS analysis, the results show that the main components of the ceramic microspheres are silicon dioxide (SiO_2) and aluminum oxide (Al_2O_3). Based on these analytical results, it can be preliminarily determined that the ceramic microspheres are made of silicate ceramic. **Table 4** shows the composition of the ceramic microspheres. Their basic properties are shown in **Table 5**.

Table 4: Composition of ceramic microspheres

Element	*w*/%	at.%	Chemical compound
C K	8.13	5.54	$CaCO_3$
O K	51.50	59.17	SiO_2
Na K	5.48	4.38	–
Al K	9.22	14.11	Al_2O_3
Si K	25.67	16.8	SiO_2

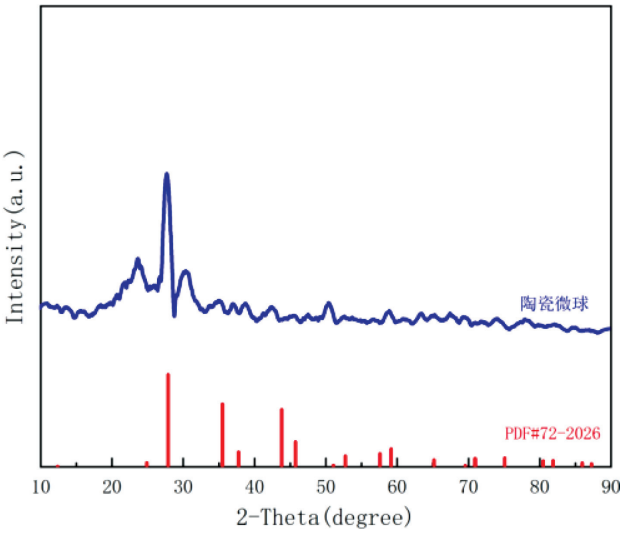

Figure 8: XRD pattern of ceramic microspheres

Table 5: Basic properties of ceramic microspheres[14]

Property	Numerical value
Particle diameter, μm	4–24
Whiteness, %	≥ 95
pH value	9.0–12.0
Mohs hardness	7
Softening point, °C	1020
Refractive index	1.53
Thermal conductivity, ($W \cdot m^{-1} \cdot K^{-1}$)	2.3
UV transmittance, mm	0–250
Oil absorption index	28

Figure 9 shows the infrared spectrum of the epoxy-resin porous sound-absorbing material. It can be seen that there is a distinct broad peak between 3500–2500 cm^{-1}, corresponding to the O-H stretching vibration peak, indicating the presence of hydroxyl groups in the sample. The absorption peak at 1546 cm^{-1} is the C=O stretching vibration peak. Since its wavenumber is less than 1700 cm^{-1}, it suggests possible conjugation of the hydroxyl group. The peak at 1474 cm^{-1} is the benzene ring skeleton vibration peak, indicating the presence of epoxy resin, forming the corresponding absorption peak. The absorption peak at 965 cm^{-1} is the O-Si stretching vibration peak, indicating the presence of silicon dioxide in the specimen. The broad peak at 3667 cm^{-1} is the N-H stretching vibration peak, resulting from the use of 593 curing agent containing diethylenetriamine, forming the corresponding absorption peak. The presence of hydroxyl functional groups on the material surface allows for better mixing between ceramic microspheres and the polymer, potentially further enhancing the material's strength.[15]

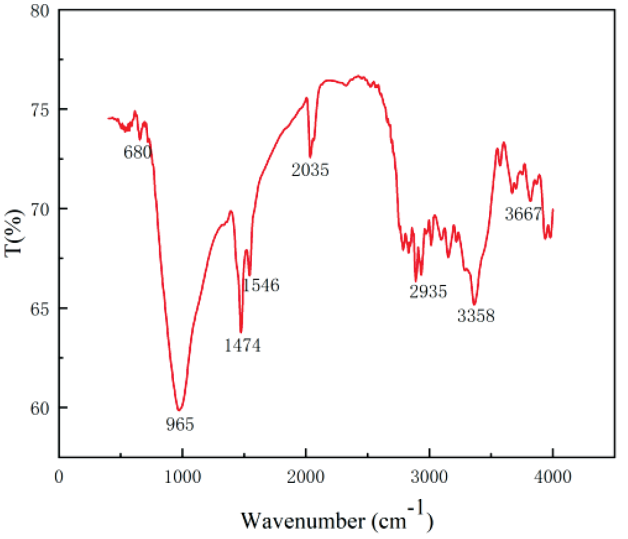

Figure 9: Infrared spectrum of epoxy-resin porous sound-absorbing material

The mass-volume direct calculation method,[16] known for its simplicity and widespread use, was employed. The density of a dense sample corresponding to the optimal specimen was tested, yielding a calculated density of 1.145 g/cm^3. Five samples from the same group were tested using the mass-volume method to determine the porosity. **Table 6** shows their porosity. The results indicate an average porosity of 71.5 % for this material. This result demonstrates a large amount of void space within the material, significantly impacting its sound absorption and microstructure.

Table 6: Porosity of the samples

Sample	Porosity, %	Average porosity, %
1	69.7	71.5
2	70.6	
3	73.6	
4	71.2	
5	72.4	

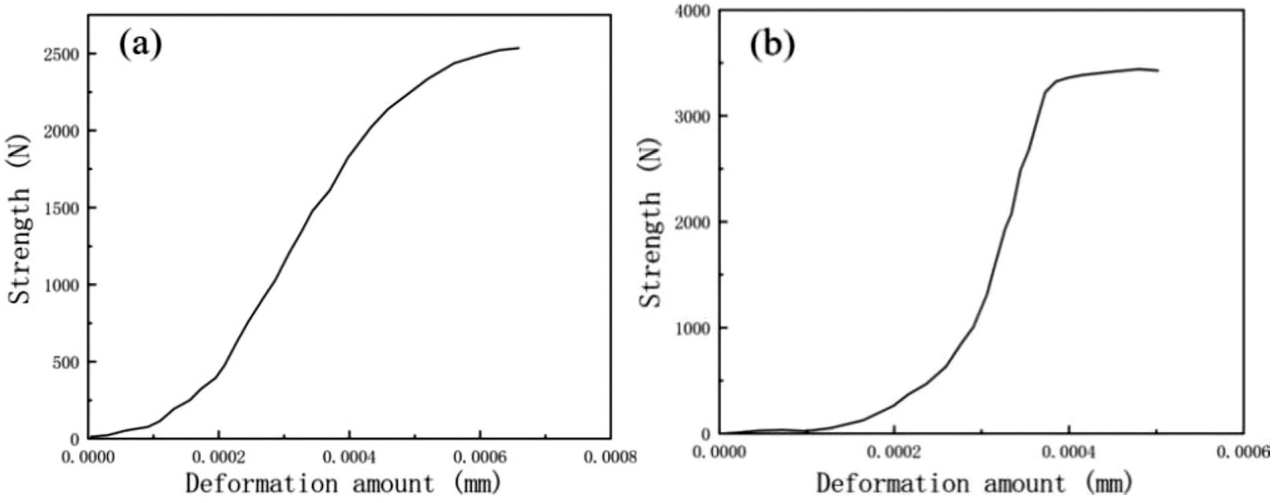

Figure 10: Pressure resistance test chart of epoxy-resin porous sound-absorbing materials: a) EM-0, b) EM-20

3.2 Physical characteristics analysis of sound-absorbing material

(1) Compressive and tensile properties analysis of porous sound-absorbing material

Figure 10 shows the compressive performance test charts of the epoxy-resin porous sound-absorbing material. **Table 7** lists the maximum compression force and compressive strength of EM-0 and EM-20 during testing. From **Figure 10**, it can be seen that under the same deformation, the force for epoxy-resin material EM-20 with 20 % ceramic microspheres is greater than that of EM-0 without microspheres. After the deformation reaches 0.0002 mm, the difference between them rapidly increases. EM-20 exhibits a larger maximum compression force and compressive strength, indicating that the addition of ceramic microspheres enhances the compressive performance of the material. This is attributed to the uniform dispersion of ceramic microspheres in the colloid, further promoting self-crosslinking of the epoxy resin, thereby improving compressive strength. **Figure 11** shows the tensile performance test charts of the epoxy-resin porous sound-absorbing material. **Table 8** lists the elastic modulus and tensile strength of EM-0 and EM-20 during testing. From **Figure 11**, it can be seen that the stress of EM-20 is significantly higher than that of EM-0, being consistent with the tensile strength test results. However, the figure also clearly shows that EM-20 fractures earlier than EM-0. This is because EM-0, without ceramic microspheres, although having lower internal structural strength than EM-20, can withstand deformations exceeding 30 % without fracturing, thus exhibiting higher toughness.

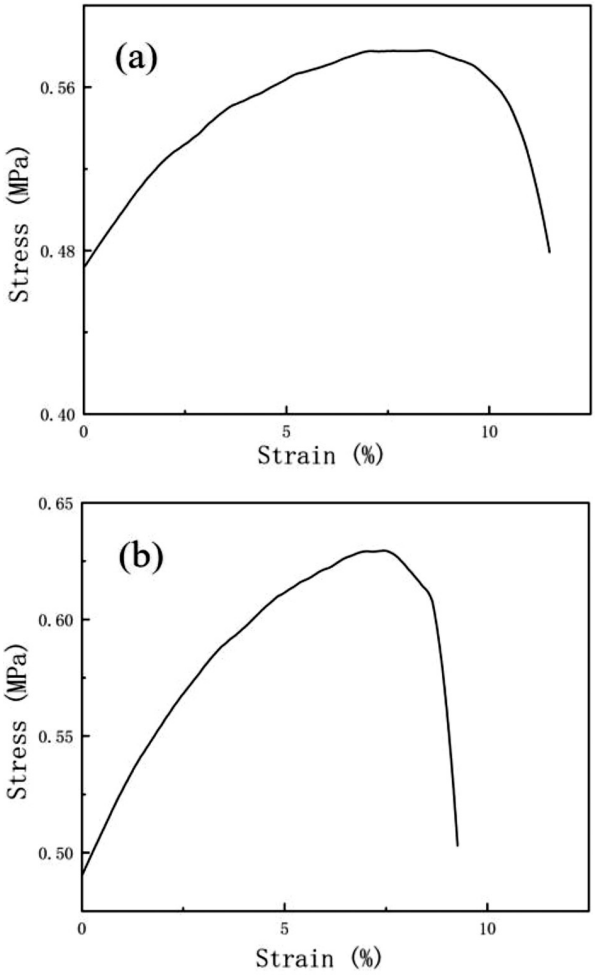

Figure 11: Tensile performance test chart of epoxy-resin porous sound-absorbing materials: a) EM-0, b) EM-20

Table 7: Maximum compression force and compressive strength of EM-0 and EM-20

Sample name	Maximum compression force, N	Compressive strength, MPa
EM-0	2130.6	0.38
EM-20	5000	0.85

Table 8: Elastic modulus and tensile strength of EM-0 and EM-20

Sample name	Elastic modulus, GPa	Tensile strength, MPa
EM-0	0.14	0.4
EM-20	0.25	0.6

(2) Abrasion resistance analysis of porous sound-absorbing material

Table 9 shows the wear of EM-0 and EM-20 during abrasion testing. Two experiments were conducted for each type of specimen. The table shows that the wear of EM-0 is 0.17 g larger than that of EM-20, indicating that the addition of ceramic microspheres enhances the material's abrasion resistance. This is because the ceramic microspheres stabilize the internal pore structure, making the overall structure less prone to damage under friction. Additionally, the high hardness of the ceramic microspheres increases the material's hardness to some extent, thereby enhancing its abrasion resistance. Further analysis of the data in **Table 9** reveals that the wear of EM-20 is only about 80 % of that of EM-0. This significant difference verifies the positive impact of ceramic microspheres on the material wear resistance. In repeated experiments, the wear rate of EM-20 consistently remained low, indicating that the addition of ceramic microspheres not only enhances the initial abrasion resistance but also helps maintain performance stability during long-term use. Besides optimizing the material's structure, ceramic microspheres, as a hard filler, increase the material's hardness and act as barriers and stress dispersers when the material is subjected to wear, significantly improving its abrasion resistance. When subjected to external friction, the high-hardness ceramic microspheres effectively resist wear, reducing scratches and indentations on the material surface. This enhanced hardness not only extends the material's service life but also allows it to maintain excellent performance under high loads or extreme working conditions.

Table 9: Wear rate of EM-0 and EM-20

Sample	EM-0, g/cm^2	EM-20, g/cm^2
1	0.186	0.038
2	0.221	0.029

(3) Water absorption of porous sound-absorbing material

This experiment determined the water absorption of the optimal specimen EM-20. To ensure accuracy and reliability, three samples from the same group were selected for testing. Through careful measurement and analysis of these samples, their respective water absorption data were collected and statistically processed. **Table 10** details the specific performance of these three samples in the water-absorption test. The calculation showed that the average water absorption rate of this group of specimens was 45.9 %. The experimental results indicate that the EM-20 specimen can absorb a considerable proportion of water upon contact, revealing that its internal structure contains a large number of pores. These pores are key factors allowing water to enter and be absorbed, and are important characteristics affecting the material's water absorption. For certain specific application fields, such as horticulture, environmental protection, or construction materials, this strong water absorption may be beneficial as it can provide good water retention or contribute to ecosystem balance. Therefore, the average water absorption rate of 45.9 % for EM-20 is an important characteristic indicator, and its advantages and disadvantages need to be evaluated in conjunction with specific application scenarios. Practical development may need to consider how to optimize the material's structure to meet different requirements.

Table 10: Water absorption rate of samples

Sample	Dry mass, g	Mass after absorption, g	Water absorption, %	Average water absorption, %
1	7.76	11.45	47.6	45.9
2	8.16	11.73	43.8	
3	8.64	12.65	46.5	

4 CONCLUSIONS

(1) The addition of ceramic microspheres improves the sound absorption of the material. However, an excessive ceramic-microsphere content blocks the internal pores of the material, thereby reducing its sound absorption. Experimental results show that the material exhibits optimal sound absorption when the ceramic-microsphere content is 20 %.

(2) The addition of ceramic microspheres significantly improves the sound insulation of the material. Comparing the EM-0 and EM-20 samples, the average sound insulation of EM-20 increased from 20.64 dB to 22.7 dB in a frequency range of 200–2000 Hz, with an average sound insulation of 21.6 dB at 2000 Hz, verifying the effective barrier effect of ceramic microspheres on sound wave propagation.

(3) According to SEM-EDS testing, ceramic microspheres are made of silicate ceramics, and their high hardness can enhance the structural stability of the material. According to infrared spectroscopy testing, the main components of the sound-absorbing material include epoxy resin and silica, and the hydroxyl functional groups on the material surface can promote the interface bonding between ceramic microspheres and epoxy resin, thereby further enhancing the strength of the material.

(4) Through physical performance testing, it is determined that the compressive strength of EM-20 is 0.85 MPa, the tensile strength is 0.6 MPa, the wear is 0.335 g/cm^2, the average porosity is 71.5 %, and the water absorption rate is 45.9 %. Therefore, the sample has a low density and is easy to transport. The most suitable amount of ceramic microspheres added is 20 %. When the addition amounts of epoxy resin, foaming agent, and ceramic microspheres are (30, 10 and 20) % of the total mass, respectively, the overall performance of the epoxy-resin porous sound-absorbing material is the best.

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