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Mechanical and sound absorption performance of addition type liquid silicone rubber reinforced with halloysite nanotubes

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Abstract

Addition-molded room temperature vulcanized liquid silicone rubber (LSR) has become the promising candidate for environmentally friendly sound-absorbing materials, but the absorption efficiency should be further improved. In this study, the Halloysite nanotubes (HNTs) are chosen as the additive with the aim to improve the sound absorption performance of the LSR. Furthermore, the effect of the addition amount of HNTs on the mechanical properties and frequency dependent sound absorption efficiencies are investigated. Results indicate that the fabricated samples show effective high-frequency sound absorption and excellent mechanical properties. The mechanism of the improved sound absorption efficiency in high frequencies is discussed, which is believed to be attributed to the synergistic effect in the multiscale structure: 1) complex interfacial interactions at the surfaces of HNTs; 2) the friction between the air in the cavity and the inner wall of HNTs. Therefore, introducing the HNTs into the traditional silicone rubber would be an efficient way to improve the absorption efficiency for high frequency sound waves.

1. Introduction

With the rapid development of industrialization, construction and consumer production, noise pollution has become one of the major problems in modern industrial fields and daily life [1–4]. Therefore, finding effective ways to reduce noise intensity has drawn much attention in material science research. At present, sound-absorbing substrates used in noise environments are mostly hard plates, including sound-insulating wooden panels [5], sound-absorbing glass panels [6], and sound-absorbing metal panels [6]. Such sound-absorbing materials can be adapted to suitable sound-absorbing panels according to the noise frequency, which could meet the noise reduction requirements of different environments. However, the installation and winding process of these hard plates is more complicated and takes up a lot of space, leading to limited applications of such hard plates [1]. Therefore, it is extremely necessary to prepare a sound-absorbing material with a wide range of sound absorption and easy implementation.

Polyurethane (PU) foam and polyvinyl chloride (PVC) foam are currently the two most common materials used for indoor sound absorption and noise reduction [7, 8]. During the preparation and processing of PU foams, the two raw materials involved in the reaction (isocyanate and combined polyether) will volatilize toxic gases. The monomer vinyl chloride (VCM) used to polymerize PVC is carcinogenic, and the auxiliary material for processing, such as plasticizers, antioxidants, etc, are also toxic [7, 9, 10]. Apparently, these two types of foam materials would inevitably bring environmental harm during the production process and consequently have an effect on human health. Therefore, the preparation of a green and environmentally friendly sound-absorbing material is extremely necessary for the development of sound-absorbing materials.

Addition-molded room temperature vulcanized liquid silicone rubber (LSR) has the advantages of simple vulcanization process and controllable shape, which can be applied to various complex production and construction environments, and the preparation process is non-toxic and harmless, meeting the requirement of green environmental protection [11]. Generally, functional additives, such as reinforcing agents, foaming
agents, flame retardants, antioxidants, conductive fillers, high thermal conductivity fillers, etc, are mixed into the colloid before vulcanization to realize a wide variety of performances for specific applications. For instance, LSR filled with particular functional fillers has the characteristics of high mechanical strength, large elastic modulus, and good heat resistance and weather resistance in previous investigations, which could be widely used in sealing parts of precision equipment [12–17]. In addition, LSR products could also be widely used in medical supplies due to their stable performance and excellent biocompatibility without toxicity [18, 19]. Furthermore, the porous LSR prepared by introducing foaming agents shows excellent comprehensive performances of softness, light weight, high resilience rate, controllable cell size and density, easy implementation, corrosion resistance, weather resistance, etc, which is capable of applying for sound absorption and noise reduction in most indoor and outdoor environments [10, 11]. On the other hand, introducing nanoporous materials into LSR could change the structure of the composites and will consequently affect the sound absorption performance to a certain extent [6, 10, 20].

Halloysite nanotubes (HNTs) has sparked considerable interest in the fabrication of halloysite polymer nanocomposites due to its unique characteristics that combine excellent mechanical properties, tubular microstructure, biocompatibility, flame retardancy and a versatile surface chemistry [21]. Its unique tubular structure is suitable for good absorption effect on high-frequency noise due to the increased damping and frictions of sound waves [22]. In addition, halloysite nanotubes can be modified by interactions with specific functional groups or thermal processing in order to improve their performance, which can be used as sound-absorbing fillers in a variety of polymer sound-absorbing materials to realize optimized properties.

It should be noticed that LSR produced by traditional technologies generally have a closed-cell structure, which leads to relatively poor noise absorption efficiency and a narrow sound absorption frequency band, limiting its application for sound absorption and noise reduction [23]. Therefore, it is of great significance to adjust the preparation process of addition-molded LSR to improve its sound absorption performance in special sound frequency bands. Thus, in this paper, HNTs is chosen as sound-absorbing fillers, and the dissociation and dispersion of HNTs structural units would be realized through the physical blending method, to prepare the halloysite nanotube reinforced liquid silicone rubber (HNTs-LSR) with excellent high-frequency sound absorption and excellent mechanical properties. Furthermore, the effect of the addition amount of halloysite nanotubes on the sound absorption efficiency and other corresponding properties of the composites would be investigated.

### 2. Materials and methodology

#### 2.1. Materials and sample preparation

According to the formulation design in table 1, the LSR, white carbon black, hydrosilicone oil and HNTs were mixed for 20 min in a precision open mill. The 2-methyl-3-butyln-2-ol was added into the mixture during mixing, to inhibit the cross-linking process and prevent partial vulcanization of the rubber during the mixing process. After the blending system is evenly dispersed, the platinum catalyst was added and the roller pitch was set to continue mixing for 10 min. After the catalyst is evenly dispersed in the system, the blend is placed in the mold, rolled evenly and sent to the vacuum drying oven. An iron plate is covered on the mold to perform constant pressure vulcanization (24 N, 150 °C, 20 min). A secondary vulcanization process (Remove the LSR from the mold and expose it to a vacuum drying oven to continue heating at 150 °C for 20 min) was conducted to prevent the incomplete vulcanization of the edge of the silicone rubber.

#### 2.2. Measurement of physical properties

##### 2.2.1. Structural characterization

The cross-section morphology of the sample was photographed by the Sony DSC-HX400 camera, and internal microstructure of the samples was characterized via the scanning electron microscopy (SEM, JSM-6460LV, Japan Electronics Co. Ltd). The FTIR spectra of HNTs and HNTs-LSR composites were analyzed using Nicolet iS10 FTIR spectrometer in the region range of 4,000 to 400 cm⁻¹. The apparent density was tested by the sealing wax method according to GB/T 6342–2009, and a group of silicone rubber samples are cut and measured many times, and the average value is finally taken as the apparent density of the specific sample.

| Component | LSR | White carbon black | Hydrosilicone oil | HNTs/phr | 2-methyl-3-butyln-2-ol | Platinum catalyst |
|-----------|-----|---------------------|-------------------|----------|------------------------|------------------|
| Amount/g | 50  | 15                  | 1.5               | 5°       | 25                     | 0.008            |
|           |     |                     |                   |          |                        | 0.09             |
2.2.2. Mechanical properties tests
The falling rebound rate was tested according to GB/T 6670–2008, and the Shore O hardness was calculated from the hardness values of 5 different positions of the sample according to GB/T 531–1999. The tensile strength and the elongation at break were tested according to GB/T 528–82.

2.2.3. Sound absorption property tests
The sound absorption properties of samples were tested using standing wave tube (AWA1622, Hangzhou Aihua Instrument Co. Ltd) according to GB/T 18696.1–2004. The sound absorption coefficients of the samples were measured at 125−5000 Hz, and the arithmetic average of the sound absorption coefficient at the six sound frequencies of 125 Hz, 250 Hz, 500 Hz, 1000 Hz, 2000 Hz and 4000 Hz is calculate to present the average sound absorption coefficient (\(\bar{\alpha}\)) of the material.

3. Results and discussion

3.1. Microstructure characteristics
Figure 1 represents the FTIR spectra in the wavenumber range of 4,000−400 cm\(^{-1}\) of pristine HNTs and LSR containing 15 phr of HNTs. For the pristine HNTs (see the black line), the adsorption around 3,685 is specifically assigned to the vibration of hydroxyl groups which are the stretching vibration of inner surface hydroxyl group. The adsorptions around 1008 and 909 cm\(^{-1}\) are specifically assigned to the OH deformation of inner-surface hydroxyl groups and inner hydroxyl groups of Al–OH librations, respectively. Compared with the pristine HNTs, the adsorptions around 2062, 1410, 1257 and 784 cm\(^{-1}\) are associated with stretching vibrations of methyl C-H, C = C, Si-CH\(_3\) and Si–C bonds, respectively. This increase of these wavenumbers indicates the interaction and agglomeration of HNTs by LSR [24].

HNTs are generally long and straight needle-like structures with a length of mostly 1 \(\mu\)m as shown in figure 2(a), and an obvious agglomeration phenomenon caused by nanometer size can be observed. The apparent color of natural HNTs nanotubes is pink due to the color of Fe\(_2\)O\(_3\) in HNTs. Thus, with increasing added amount of HNTs, the apparent color of the sample changes from light brown to dark brown. When the added amount of HNTs is below 25 phr, all HNTs can be finely dispersed in the LSR. As shown in figure 2(b), the sample with 30 phr added amount of HNTs has obvious delamination, which should be attributed to the agglomeration effect caused by the excessive addition of HNTs. From the SEM photos shown in figures 2(c) and (d), the black region is detected to be the LSR matrix, the white region is the HNTs agglomerate, and the gray region is combination phase of HNTs and LSR. Obviously, the sample with 15 phr HNTs addition shows relatively more uniform dispersion effect than that of the sample with 25 phr addition, indicating that the dispersion effect would be obvious with increasing addition amount of HNTs, which could be the result of the enhanced agglomeration phenomenon of HNTs in the LSR. Therefore, the composite structure of the HNTs-LSR could be significantly influenced by the addition amount of the HNTs.
3.2. Mechanical properties

Table 2 summarizes the apparent densities of all samples and the corresponding basic elastic performances. It is obvious that the apparent density would be increased with large addition amount of the HNTs into the LSR, but the increasing rate is less than 32% for all samples, which indicates that adding HNTs would not largely affect the porosity of the sample. With the increasing addition amount of HNTs, the Shore O hardness of the samples first and reaches the largest value of 49.6 HO with 15 phr HNTs addition, then the Shore O hardness decreases with large amount addition of HNTs. This non-linear change in Shore O hardness could be attributed to the dispersion performance of HNTs in LSR: HNTs should be uniformly dispersed in the colloid after repeated physical blending, but excessive concentration (larger than 15 phr) of HNTs in LSR would result in agglomeration and poor uniformity, leading to uncoated HNTs in the composite. These agglomerations of uncoated HNTs in LSR would influence the internal force determined by the relative slip between HNTs, which would result in the reduction of the hardness. Since the elastic properties of HNTs-LSR composites is mainly contributed by the LSR, increasing the adding amount of HNTs would reduce the recovery force of silicone rubber after deformation, and falling rebound rate of samples decreases with large addition amounts of HNTs.

Table 2. Physical properties of HF–LSR under different amounts of HNTs added.

| Sample ID | HNTs phr | Apparent density g/cm³ | Shore O hardness HO | Falling rebound rate % |
|-----------|----------|------------------------|---------------------|-----------------------|
| 1         | 0        | 0.6303                 | 46.0                | 68.0                  |
| 2         | 5        | 0.7865                 | 47.8                | 66.0                  |
| 3         | 10       | 0.8089                 | 48.4                | 62.4                  |
| 4         | 15       | 0.8103                 | 49.6                | 56.0                  |
| 5         | 20       | 0.8295                 | 41.5                | 44.0                  |
| 6         | 25       | 0.8311                 | 34.0                | 36.0                  |

Figure 2. Structure characteristics of the samples. (a) SEM of HNTs; (b) Photo of the HNTs-LSR sample with 30 phr addition of HNTs; (c) SEM of the HNTs-LSR sample with 15 phr addition of HNTs; (d) SEM of the HNTs-LSR sample with 25 phr addition of HNTs.
should also be noticed that the dispersion effect of HNTs in the colloid is better when the addition amount of HNTs is less than 15 phr, and the elasticity of the sample shows an approximately linear decreasing; when the addition amount is higher than 15 phr, the elasticity of the sample is greatly reduced with increasing adding amount of HNTs, since the uncoated HNTs are agglomerated in the LSR, which would destroy the resilience of the composite.

The tensile strength and the corresponding tensile strength and elongation at break of HNTs-LSR composites with different addition amount of HNTs are plotted in figure 3. With the increasing addition amount of HNTs, the tensile strength first increases gradually and then decreases drastically with the further increase of HNTs addition, and the maximum tensile strength (0.84 MPa) is achieved with 15 phr addition of HNTs into LSR. This improved tensile strength should be the result of physical cross-linking via hydrogen bonds, which are formed from the surface Si-OH of the HNTs tube and the Si-O-Si of the silicone rubber molecular chains. In addition, HNTs have the long straight tubular structure and specific orientation, which would result in strong restrictive effect on the silicone rubber as the result of the physical entanglement with silicone rubber molecular chain. However, with the large addition amount of HNTs (> 15 phr), the agglomeration effect would be prominent due to the nano-effect, which would lead to much more structural defects and consensually reduce the tensile strength of the composites.

The elongation at break of HNTs-LSR composites decreases monotonically with the increasing addition amount of HNTs and the reducing rate would be enlarged with 10–20 phr HNTs addition. Micron-sized HNTs with certain orientations are randomly dispersed in the LSR matrix after physical blending, which would limit the movement of molecular chains in LSR, since these HNTs cannot accomplish large deformations accompanied by silicone rubber, leading to the reduced elongation at break of the composites. When the amount of HNTs added is less than 15 phr, the more HNTs added into silicone rubber, the stronger the restriction of HNTs to silicone rubber molecular chains. The less deformation would result in continuous decreasing of the elongation at break. It should also be noticed that structural defects would be increased significantly due to the aggregation effect of HNTs when the addition amount of HNTs is higher than 15 phr, which would reduce the effective area of the tensile force on the experimental sample section, leading to the stress concentration and reduce the elongation at break in another way.

3.3. Sound absorption properties
It has been well established that the sound absorption coefficient of the material is a function of frequency, and unlike common porous materials that the sound absorption would generally increases with frequency increasing, the fabricated HNTs-LSR composites shows fluctuational sound absorption coefficient changing with increasing frequency as shown in figure 4.

It is obvious that the sound absorption coefficient of the HNTs-LSR composites show different changing patterns in low (0–3000 Hz) and high (3000–5000 Hz) frequency ranges. In the frequency range of 125–500 Hz, neither the LSR nor HNTs-LSR composite shows obvious sound absorption performance, which should be the result of wave-passing behavior caused by the scattering effect for long wavelength. At the sound frequency of 750–2000 Hz, the sound absorption coefficient of the sample is lower than that of LSR. This reduced sound absorption efficiency of HNTs-LSR composites should be attributed to the improved scattering effect for long wavelength, since the HNTs, white carbon black and LSR could all serve as inhomogeneous scattering mediums.
which would result in much more fraction of long wave scattering from the edge of samples, and consequently reduce the sound absorption efficiency in middle frequency range. This reduced absorption induced by the enhanced scattering effect of the composites would become progressively less obvious with further increasing the sound frequency, and the sound absorption coefficients of HNTs-LSR composites are significantly improved, and the increment is enlarged with increasing addition amount of HNTs. It could be clear seen that the largest absorption coefficient is achieved at 4000 Hz with 25 phr addition of HNTs in the composite. This improved absorption efficiency in high frequency range could be attributed to the sound energy loss caused by the friction effect between the intrinsic walls of HNTs and the incident sound waves. High frequency sound wave would be easier to penetrate into the cavity and cause the air vibration, which would consume the sound energy via friction. On the other hand, a certain speed gradient would also cause the energy loss when transmitting at different interfaces between LSR and HNTs nanotubes, since the propagation speed of sound waves in different media is different. In addition, HNTs nanotubes are expected to be randomly dispersed in the LSR matrix, and they would be presenting three positional relationships (perpendicular, parallel and oblique) to the direction of incident sound wave, which would cause the sound wave to be reflected in other directions when it propagates to the HNTs wall, making the sound wave more dispersed and reducing the sound energy in a particular direction. Therefore, with additive HNTs into the LSR, the sound absorption efficiency shows different trends in different frequency range, and the sound absorption performance of the HNTs-LSR composites would be improved at high frequencies.

To further specify the improved sound absorption efficiency of the HNTs-LSR composites in the high frequency range, the sound absorption coefficients of all samples at 3000 Hz, 4000 Hz and 5000 Hz are plotted in figure 5. At 3000 Hz, the difference between the absorption coefficients of all samples is not significant, since the synergy between the edge scattering effect and the energy dissipation of HNTs. With the incident wave of 4000 Hz and 5000 Hz, the edge scattering effect becomes non-significant with relative shorter wavelengths, and the energy dissipation caused by the HNTs becomes the key factor to improve the sound absorption efficiency: at 4000 Hz, the sound absorption coefficient of HNTs-LSR composite with 25 phr HNTs addition reaches the maximum value of 75.8%, which achieve a 42.7% increase relative to than of the LSR sample; at 5000 Hz, the sound absorption coefficient composite with 25 phr HNTs addition is nearly double that of the LSR sample. Furthermore, the average sound absorption coefficients of all samples in this sound frequency range are calculated and plotted in figure 5(d), in order to evaluate the comprehensive sound absorption performance of HNTs-LSR composites in the high frequency range. The average sound absorption coefficient of HNTs-LSR composites increases with larger addition amount of HNTs. This is because high-frequency sound waves have large energy and weaker wave diffraction, and the sound waves are more likely to penetrate into the internal cavity of HNTs and cause vibration of the air. As the addition amount of HNTs increases, the structure of the cavity formed by nanotubes is more obvious, and the air in the cavity increases correspondingly. Thus, the friction between the air in the cavity and the inner wall of the HNTs would be enhanced to reduce sound wave energy. When the addition amount of HNTs is 25 phr, the average sound absorption coefficient of HNTs-LSR in the high frequency range from 3000 to 5000 Hz reaches the maximum value of 51.8%, which is 54.2% higher than 33.6% of the pristine LSR.

![Figure 4. Frequency dependent sound absorption coefficient of HNTs-LSR composites with different addition amounts of HNTs.](image-url)
Furthermore, the effect of additive HNTs into the LSR on the sound absorption performances could be explained via interactions between sound waves and composite structures. HNTs chosen as the nano-fillers are tubular with a pore size of 15–20 nm, which have extremely low flow resistance. Thus, the propagating speed in the HNTs shows different characteristics comparing to that in the flexible LSR matrix. As shown in figure 6(a), complex interfacial interactions would occur at the surface of the composite: reflection, transmission and scattering with finite volume. The increased addition amount of HNTs would change the apparent density, which would significantly influence the acoustic impedance of the composite and determine the proportional

![Figure 5. Effect of the addition amount of HNTs on sound absorption coefficient of HNTs-LSR composites in the high frequency. (a) 3000 Hz; (b) 4000 Hz; (c) 5000 Hz; (d) Average values in 3000–5000 Hz.](image)

![Figure 6. An illustration for interaction of sound waves with the HNTs-LSR composites.](image)
distributions of the energy of the reflected, transmitted and scattering waves. And this change in the transmitted wave into the composites would be the pre-constraint factor for the sound absorption characteristics of the composites. During the propagation in the composites, the sound wave would interact with the dispersed HNTs nanotubes in different contacting mode as shown in figure 6(b): (1) When the propagation direction of sound waves is perpendicular to the wall of HNTs, a small part of the sound wave perpendicular to the axis of the HNTs would penetrate into the tubes, which would cause the vibration of the air in the tubes to induce the fraction and consume the sound energy. The most part of the propagating sound wave would be reflected at the curved surface of the HNTs, resulting in the enhanced scattering effect of the sound wave to reduce the sound wave energy in incident direction. (2) When the propagation direction of sound waves is parallel to the wall of HNTs, the sound wave would directly propagate into the tubes without any reflection, and cause the vibration of the air particles in the tube cavity, which would consume the sound energy via the friction between the air and the tube inner walls. (3) When the propagation direction of sound waves is oblique to the wall of HNTs, the most of the sound wave would directly contact the curved surface of the curved surface of HNTs and result in a scattering-like reflection to reduce intensity of the propagating sound wave. A very small part of the sound wave could be transmitted into the tube, causing the internal friction between the air and the HNTs walls to reduce the sound intensity. It should be noticed that all these interactions between sound waves and composite structures are frequency dependent. In the low frequency range, the sound wave is more easily diffracted to other directions through the surface of the structures due to the larger wavelength, and internal friction between the air in the nanotubes and the HNTs walls would be relative inapparent. With increased sound frequency, the diffraction effect is reduced, and the sound waves are more easily to be transmitted into the cavity of the HNTs and excite the vibration of the air in the cavity. Therefore, the fabricated HNTs-LSR composites have a poor absorption effect for low frequency noise, but have a better absorption effect for high frequency sound waves.

4. Conclusion

With the aim to improve the sound absorption efficiency of the LSR, the HNTs that could qualify for efficient absorbing in high frequency range due to their particular tubular structures, are chosen as the additive into the LSR composites. The HNTs-LSR composite samples were prepared via the physical blending method and the effect of different addition amount of HNTs on the microstructure, mechanical and frequency dependent sound absorption performances were investigated. Based on the obtained results, the following conclusions can be drawn:

(1) The HNTs-LSR composites could be successfully prepared by the physical blending, and the HNTs could be uniformly dispersed in the LSR matrix when the addition amount is less than 25phr. Large addition amount of HNTs could cause obvious agglomerations of halloysite nanotubes, which would result in different influences on the mechanical and sound absorption performances.

(2) Introducing HNTs as reinforcements would improve the mechanical properties the composites. The tensile strength as well as the Shore O hardness all first increase gradually and then decreases drastically with the increasing addition amount of HNTs, and the largest tensile strength (0.84 MPa) and Shore O hardness (49.6 HO) are achieved with 15 phr HNTs addition, respectively. This degradation of the tensile strength and hardness should be attributed to the increased structural defects caused by the enhanced agglomeration.

(3) The elastic properties, including falling rebound rate and elongation at break, both decreases with increasing addition amount of HNTs, which should be the result of the enhanced restriction effect of HNTs to silicone rubber molecular chains.

(4) With additive HNTs into the LSR, the sound absorption performances of HNTs-LSR composites are decreased for low and middle (<3000 Hz) sound wave, and improved for high frequency (> 3000 Hz) sound wave. The largest absorption coefficient (75.8%) is achieved at 4000 Hz with 25 phr addition of HNTs.

(5) The improved sound absorption efficiency of the HNTs-LSR composites in high frequencies should be the result of the synergistic effect between complex interfacial interactions at the surfaces of HNTs and the friction between the air in the cavity and the inner walls of HNTs.
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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

Conflict of interest

The authors declare that they have no conflict of interest.

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