

Effect of modification of rhamnolipids on barium titanate powder and the mechanical properties of PHBV-BTO composites

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ABSTRACT: In this study, the mechanical properties of poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV)/barium titanate (BTO) composites were improved by the surface modification of BTO powder with rhamnolipid (RL) via a wet chemical method. Fourier transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), Brunauer Emmett Teller (BET) surface area analysis, water contact angle measurement, scanning electron microscopy (SEM), and thermogravimetric analysis (TGA) were carried out. The water contact angle rose from 0° of unmodified BTO to 125° of RL-modified BTO (m-BTO). FTIR analysis confirmed that RL molecules were immobilized on BTO particle surfaces, primarily through hydrogen bonding and van der Waals forces. XPS characterization showed that BTO and RL have enhanced interfacial interactions, but these interactions are non-covalent. BET results showed that increasing the RL dosage caused a gradual decrease in the specific surface area and adsorption capacity of m-BTO. Energy dispersive spectrometer (EDS) analysis further showed a pronounced decrease in carbon content and concurrent increases in Ba and Ti fractions, indicating effective removal of impurities and successful adsorption of RL. TGA revealed a reduced mass loss for m-BTO, confirming improved thermal stability due to surface coating. SEM observations showed that RL modification prevents BTO particle agglomeration and improves particle dispersion in PHBV matrix. Notably, the optimal m-BTO loading resulted in a 32.7% improvement for the mechanical properties of PHBV/m-BTO composites compared to unmodified PHBV/BTO. This work presents a green and efficient surface modification strategy for BTO fillers, which offers good prospects for the fabrication of high-performance biomedical piezoelectric composite scaffolds.

KEYWORDS: biosurfactant, rhamnolipid, barium titanate powder, surface modification, poly(3-hydroxybutyrate-co-3-hydroxyvalerate)

INTRODUCTION

Barium titanate (BaTiO_3 , BTO) is a high-performance inorganic functional material with high dielectric constant and low dielectric loss, so it has a wide range of applications in electronic, multilayer capacitors, positive temperature coefficient (PTC) thermal components, and biomedical engineering [1–3]. In biomedical applications, BTO particles are usually blended with organic polymers to make functional devices, like piezoelectric cartilage repair scaffolds and flexible bioelectronic devices [4–7]. But pristine BTO powder has surface hydroxyl groups, so it has hydrophilicity. The pristine BTO and hydrophobic polymer matrices have weak interfacial bonding strength, which leads to severe particle agglomeration, restricting dispersion uniformity and final performance of BTO composites. Therefore, surface modification for BTO powder is essential to overcome these limitations.

Traditional surface modifiers for BTO, such as silane coupling agents and synthetic polymer modifiers, have inherent problems including cytotoxicity, non-biodegradability, and bad effects on composites' dielectric properties, which makes them hard to use in biomedical fields. While, biosurfactants have been

extensively studied in biomedical material research because they are environmentally friendly, have low toxicity, and are very biocompatible. As a glycolipid biosurfactant, rhamnolipid (RL) has a superior surface activity, full biodegradability, and biological safety, so it's a good modifier for inorganic material surface functionalization [8, 9]. The unique amphiphilic molecular structure of RL can form uniform organic coating films on inorganic particle surfaces [10–12], which improves the dispersion stability, corrosion resistance, and oxidation resistance of modified materials [13–16]. Therefore, RL-modified BTO has a great application potential in biomedical piezoelectric scaffolds and flexible electronic composites.

Although RL has superior properties, there is still a lack of research on RL-modified BTO for PHBV biocomposite applications. The relationship between the structure and activity of RL surface modification and the mechanical performance of PHBV/BTO composites is still not clear. In this paper, RL was used to modify BTO powder by a wet method to lower surface energy and enhance the dispersion performance of BTO particles in PHBV matrices. The hydrophobicity of m-BTO was analyzed by measuring the water contact angle. XPS, FTIR, and EDS were applied to

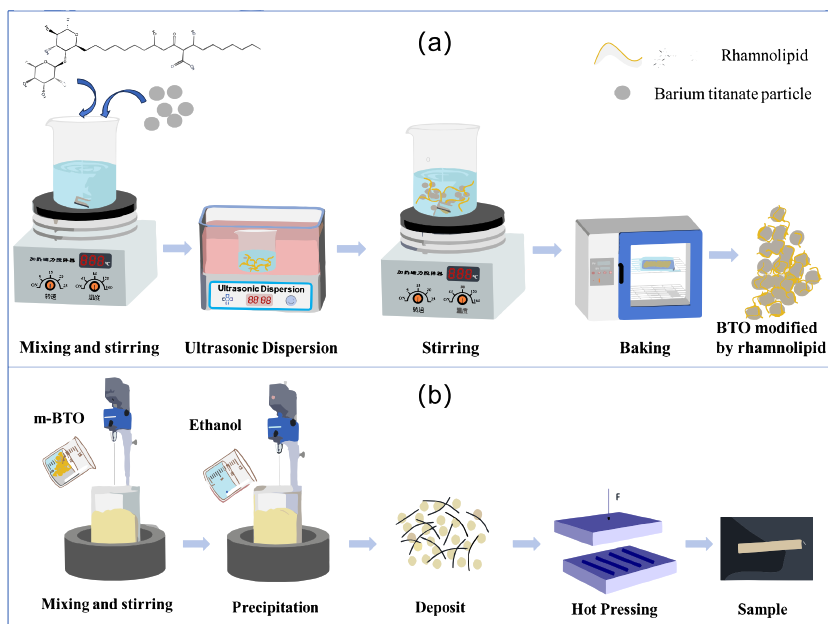


Fig. 1 The schematic of (a) the fabrication of BTO modified by RL and (b) the fabrication of PHBV/m-BTO composites.

analyze the surface elemental composition, chemical states, and variations in functional groups of the modified particles. Additionally, BET tests were utilized to characterize the surface area changes. SEM was used to examine the micro-morphology and dispersion state of m-BTO in PHBV composites. The mechanism behind the surface property improvement due to RL modification was analyzed. Moreover, the advantages and limitations of RL modification were clarified by comparing it with conventional modification methods mentioned in previous studies. This study's aim is to provide a theoretical and experimental basis for the development of high-performance biodegradable piezoelectric composites.

MATERIALS AND METHODS

Materials

Barium titanate powder (BaTiO_3 , 99.5% purity) was purchased from Shanghai Dianyang Industrial Co., Ltd. (Shanghai, China). Rhamnolipids were obtained from Wuhan Zhong Biotechnology Co., Ltd. (Wuhan, China). Ethanol (analytical grade) was purchased from Xilong Science Co., Ltd. (Shantou, China). PHBV (ENMAT Y10) powder was obtained from Ningbo Tian'an Biomaterials Co., Ltd. (Ningbo, China).

Methods

Surface modification of barium titanate

The BTO surface modification schematic procedure is shown in Fig. 1(a). The solid-liquid mass ratio of BTO powder to ethanol was set to 1:20. RL was added to the BTO-ethanol suspension at mass fractions of 0–5 wt% relative to BTO powder. The mixed suspension was

ultrasonically treated for 10 min to get preliminary dispersion, then continuously stirred at 50 °C for 50 min to finish the surface modification reaction. After the reaction, the suspension was filtered, washed with ethanol to remove unadsorbed RL, and then dried at 60 °C for 12 h to get RL-modified BTO powder (m-BTO).

Composite preparation

The fabrication process of PHBV-based composites is depicted in Fig. 1(b). Pristine BTO or m-BTO filler was dispersed in chloroform (20 wt% relative to PHBV), and then PHBV powder was added. The mixture was mechanically blended for 1 h to create a homogeneous suspension. After the solvent was completely evaporated at 50 °C, the composite precursor was pressed hot at 180 °C and 10 MPa for 10 min to get target composite samples. In this study, the preparation process was not just referenced by previous research [17], but was independently optimized.

Surface characterization and test

XPS (Thermo K-Alpha, Thermo Fisher Scientific, Waltham, USA) was utilized to analyze the surface elemental composition and chemical bonding states of BTO and m-BTO. FTIR and attenuated total reflection FTIR (ATR-FTIR, Thermo IS50, Thermo Fisher Scientific) were used to determine the surface functional group changes before and after modification. The surface hydrophobicity of BTO particles was analyzed using a water contact angle measuring instrument (SDC-350E, Shengding Precision Instrument Co., Ltd., Dongguan, China). For the powder contact angle testing, BTO and m-BTO powders were pressed into uniform pellets with a diameter of 13 mm and a pressure

of 10 MPa. A 5 μ l deionized water droplet was placed on the pellet surface, and sessile drop method was used to measure the contact angle at 25 °C. Each sample was tested in five parallel replicates, the results were presented as mean $\pm$ standard deviation (SD).

The specific surface area and pore structure of the samples were analyzed using the BET analysis method (ASAP 2460, Micromeritics, Norcross, USA). SEM (FEI Phenom Prox, Eindhoven, Netherlands; ZEISS, Sigma 300, Oberkochen, Germany) was used to observe the microscopic morphology and particle dispersion of composites. The bending mechanical properties of composites were tested according to the national standard GB/T 9341-2008. The specimen has a size of 80 mm $\times$ 10 mm $\times$ 4 mm, and there are five parallel samples for each group. The test data were presented as mean $\pm$ SD. One-way analysis of variance (ANOVA) combined with Tukey's post-hoc test was performed for statistical significance analysis of bending strength data, and $p < 0.05$ was defined as a statistically significant difference.

RESULTS AND DISCUSSION

Analysis of the surface modification effect of barium titanate powder

XPS characterization was done to check whether the successful adsorption of RL on BTO surfaces was achieved, and analyze the interfacial interaction between RL and BTO. The XPS full spectra of pristine BTO and m-BTO are shown in Fig. 2(a). Comparing with unmodified BTO, m-BTO shows a significant increase in the surface carbon and oxygen contents. This preliminary evidence supports the successful immobilization of RL organic molecules on BTO particle surfaces [18, 19].

For detailed peak analysis as follows: the characteristic peaks at 780 eV and 794 eV correspond to Ba 3d_{5/2} and Ba 3d_{3/2} of Ba²⁺ in BTO, respectively [20, 21] and the binding energy of the Ba 3d core-level transition is centered at approximately 796 eV [22]. The peaks at 458 eV and 464 eV are attributed to Ti 2p_{3/2} and Ti 2p_{1/2}, which correspond to the Ti⁴⁺ oxidation state of TiO₆ octahedrons in the BTO perovskite structure [23, 24]. The peak at 530 eV represents lattice oxygen derived from Ti–O and Ba–O chemical bonds in BTO crystals [25]. For m-BTO, the newly emerged peak at 285.0 eV originates from C–C and C–H bonds of hydrophobic alkyl chains and sugar ring structures in RL molecules [26, 27]. The peak at 286 eV is assigned to C–O–C glycosidic bonds and hydroxyl (–OH) groups in RL [28, 29], while the peak at 532 eV corresponds to ether bond and glycosidic bond oxygen atoms in RL molecular chains [30].

When the RL dosage was more than 2 wt%, we observed a distinct shift in the O1s characteristic peak. This indicates changes in the local chemical environment of the BTO surface, and enhanced interfacial

interaction between BTO and RL [31–33]. Combined with the subsequent FTIR results, this interfacial interaction was found to be a weak intermolecular force, not a strong covalent bonding. The peak shift phenomenon indicates the redistribution of surface charge and improved interfacial adhesion, which is advantageous for stress transfer at the filler-matrix interface in composites [34]. Moreover, XPS results alone cannot confirm the formation of chemical bonds, and spectral shifts might also be attributed to physical adsorption and surface charge redistribution.

Besides XPS, diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) and ATR-FTIR tests were utilized to clarify the structural changes of BTO before and after RL modification, and the spectra are shown in Fig. 2(b,c). DRIFTS results indicate that the absorbance intensity of the Ti–O lattice vibration region (500–700 cm^{−1}) of BTO remained unchanged after RL modification, which demonstrates that RL adsorption did not damage the intrinsic perovskite crystal framework of BTO. In the 750–850 cm^{−1} region, m-BTO had a slight decrease in absorbance, suggesting that the local Ti–O chemical environment of BTO was perturbed by the RL surface adsorption. In the 90–140 cm^{−1} range, the reflectance of m-BTO was always lower than that of pristine BTO. This because the vibration absorption of organic functional groups in RL, such as C–O–C stretching (1050–1150 cm^{−1}), C–OH stretching (1050 cm^{−1}), and C–H bending (1350–1380 cm^{−1}), is responsible for the vibration absorption of organic functional groups in RL.

Comparing to DRIFTS results, ATR-FTIR spectra indicate that m-BTO has a higher reflectance than pristine BTO. This opposite trend is due to the formation of a low-refractive-index RL organic film on the BTO surface, which alters the refractive index matching state between ATR crystal and sample surface, thus increasing the reflected light intensity. Although ATR-FTIR can not directly show the absorption characteristics of functional groups, the big spectral difference between BTO and m-BTO strongly indicates the formation of a stable RL organic coating layer on BTO surface.

Combined FTIR results, it indicates that RL molecules are stably adsorbed on BTO particle surfaces, primarily due to weak hydrogen bonding and van der Waals forces, and no new covalent bonds have been generated. The complementary DRIFTS and ATR-FTIR data together confirm the successful construction of a stable RL-modified BTO hybrid interface system.

The water contact angle test results for m-BTO under various modification conditions and RL dosages are shown in Fig. 3. The optimal modification parameters were found to be a reaction temperature of 50 °C, reaction duration of 50 min, and RL dosage of 1 wt%. Under the optimal conditions, the water contact angle of BTO powder has increased from 0° (pristine BTO) to 125°. This indicates a significant transition from hydrophilicity to lipophilicity on the BTO surface. This

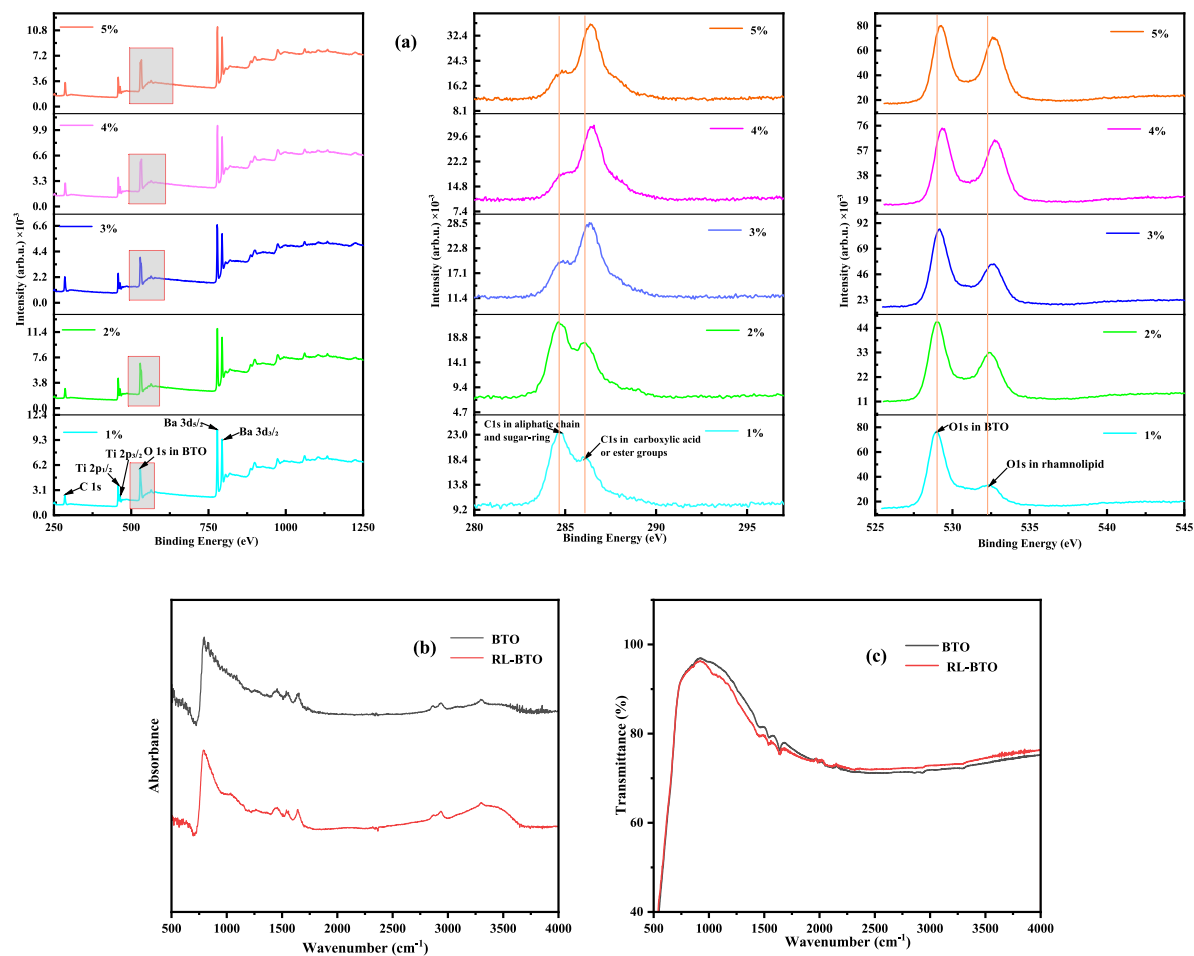


Fig. 2 Spectra of RL-modified barium titanate tested by (a) XPS, (b) DRIFTS, and (c) ATR-FTIR.

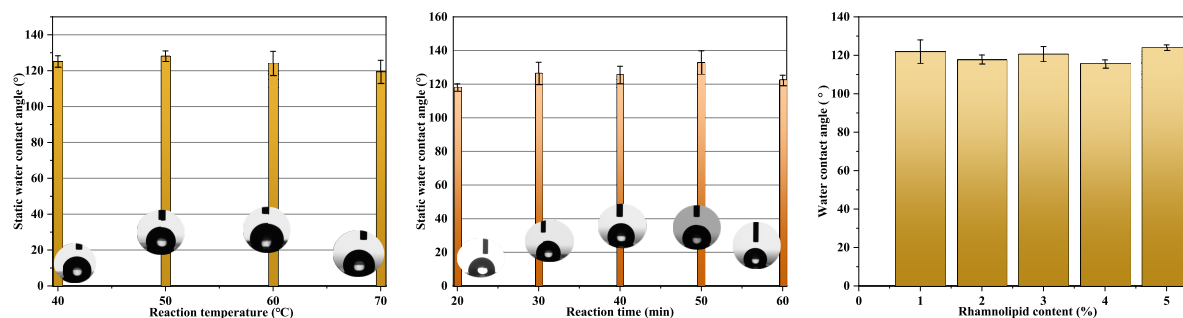


Fig. 3 Water contact angle of m-BTO with different modification conditions.

result aligns with XPS data, which shows that the hydrophobic C–C/C–H groups are enriched on the m-BTO surface.

Further increasing the RL dosage beyond 1 wt% doesn't keep the hydrophobicity of m-BTO continuously improving, so 1 wt% RL is enough to achieve saturated monolayer adsorption the BTO surface in this work. When the RL dosage is more than 2 wt%, multilayer adsorption happened on the BTO surface,

but the excess RL molecules doesn't further optimize the surface hydrophobicity. This is in line with the XPS spectral variation trend. The improved lipophilicity of m-BTO significantly reduces the surface wetting performance of BTO powder. This will enhance the interfacial compatibility with hydrophobic PHBV matrices and establish a solid foundation for the improvement of composite interface bonding strength.

The nitrogen adsorption–desorption isotherms of

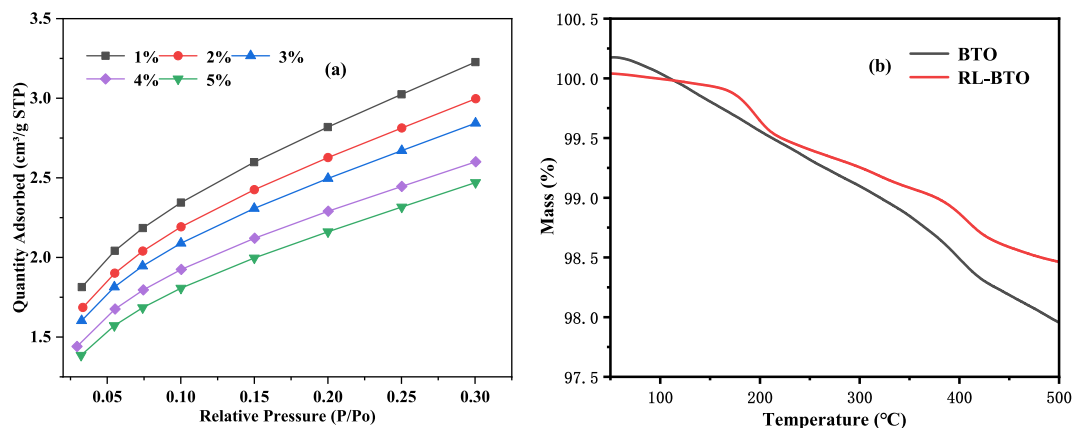


Fig. 4 Curves of m-BTO tested by (a) BET and (b) TGA.

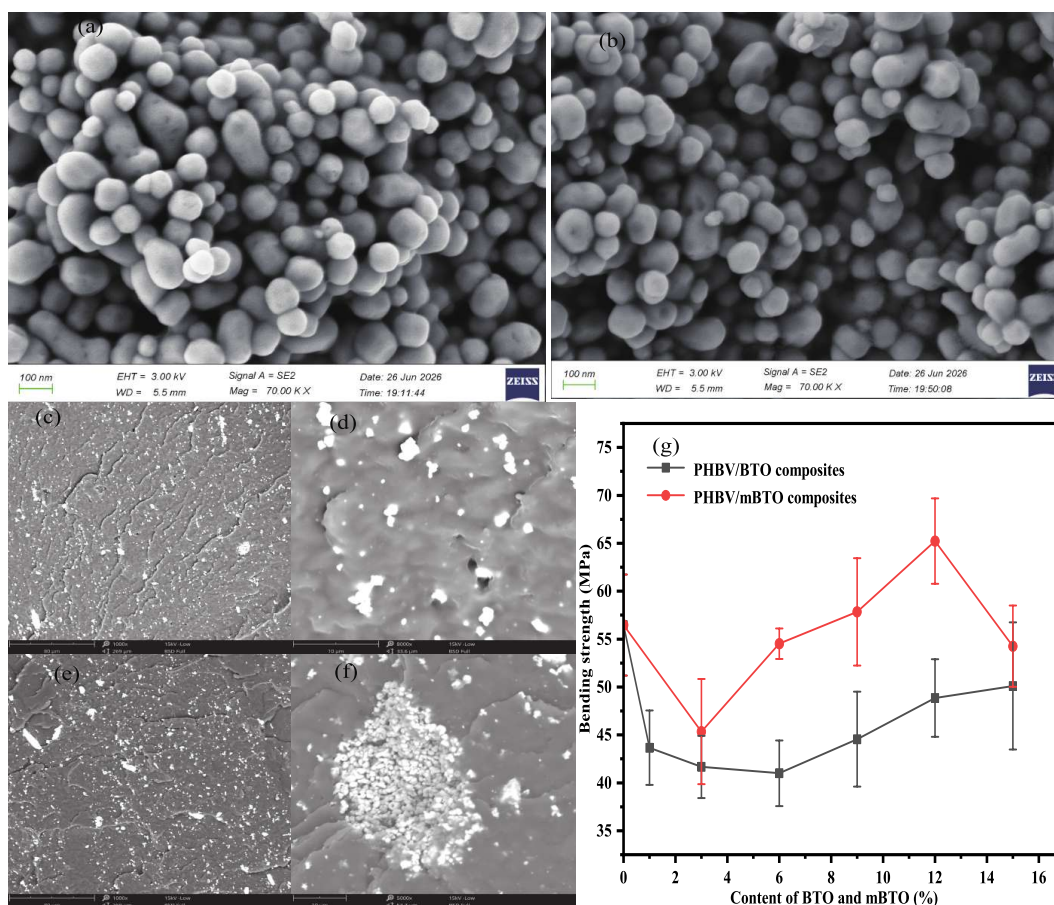


Fig. 5 SEM images of (a) BTO, (b) m-BTO (with 1% RL), PHBV/m-BTO (12 wt%) composites (c) 1000X, (d) 8000X, and PHBV/BTO (12 wt%) composites (e) 1000X; (f) 5000X, and (g) bending strength of the composites.

m-BTO with various RL dosages are depicted in Fig. 4(a), and the specific surface area data are listed in Table 2. All the modified BTO samples show nitrogen adsorption characteristics similar to typical non-porous materials. At low relative pressure, the interfacial interaction between RL and BTO particles has been

promoted, which facilitates nitrogen adsorption on the particle surface. With the rise of relative pressure, multilayer nitrogen adsorption forms, and the adsorption capacity increases continuously near the saturated vapor pressure.

With the increase of RL dosage, the nitrogen

Table 1 Assignment of the main IR absorption bands of pure BTO and RL-BTO in the diffuse reflectance spectra [35–38].

Wavenumber (cm ⁻¹)	Possible assignment
750	Ti–O lattice vibration (surface perturbation)
850	Ti–O stretching
900	Sugar ring skeleton vibration (overlap)
950	Sugar ring or C–OH bending
1000	C–O–C stretching (rhamnolipid)
1050	C–OH stretching (rhamnolipid)
1100	C–O–C asymmetric stretching
1150	C–O–C symmetric stretching
1200	C–H bending or sugar ring
1250–1400	C–H bending (~1380 cm ⁻¹) and carboxylate vibrations

Table 2 BET Surface Area of m-BTO with different RL dosages.

RL content (%)	1	2	3	4	5
BET surface area (m ² /g)	10.367	9.659	9.158	8.385	7.928

adsorption capacity and specific surface area of m-BTO decreased. The surface area of pristine BTO is 10.37 m²/g, and when the RL dosage reaches 5 wt%, it decreases to 7.93 m²/g. This phenomenon is due to the RL organic molecules' filling effect on the rough surface of BTO particles [39]. The adsorbed RL covers the micro-pits and uneven structures on the BTO surface, thereby reducing the surface roughness and specific surface area. Combining XPS, contact angle results, and the BET data, it further supports the successful coating of RL on the BTO particle surface.

TGA tests were performed to assess thermal stability of both pristine and m-BTO powder, and thermogravimetric curves are depicted in Fig. 4(b). In the 50–500 °C temperature range, the total mass loss of pristine BTO is about 3.6%, mainly because the surface adsorbed water was desorbed, and residual carbonaceous impurities were thermally decomposed. Comparing it, the total mass loss of m-BTO was 2.0%, which shows an improved thermal stability.

The m-BTO has a reduced mass loss mainly because during the modification process, thermally unstable carbonaceous impurities are removed. Moreover, the stable RL organic coating layer on the BTO surface can prevent the condensation reaction of surface hydroxyl groups at high temperatures, thereby enhancing thermal stability of modified powder. Consistent with EDS results, TGA data shows that RL modification not just purifies the BTO particle surface, but also constructs a stable organic functional layer. But, since the pristine BTO substrate has a non-negligible thermal mass loss, TGA alone can't precisely measure the adsorbed RL mass, so it needs to be supplemented by precise quantitative detection methods in subsequent studies.

Table 3 EDS results of BTO and m-BTO (with 1% RL).

Sample	C (wt%)	O (wt%)	Ba (wt%)	Ti (wt%)
BTO	24.59	22.98	36.37	16.06
m-BTO	11.01	23.06	45.47	20.46

The micro-morphologies of pristine BTO and 1 wt% RL-modified m-BTO observed by SEM are shown in Fig. 5(a,b). Pristine BTO particles have a smooth and clean surface, with sharp and distinct edge contours. The adjacent particles are independent, with clear boundary separation. For m-BTO particles, the particles have a rougher surface, which is covered with a uniform, translucent, and organic coating. The coating layer blurs the sharp edges of BTO particles, and there are inter-particle adhesion and bridging structures. The surface organic layer acts a flexible binder, binding adjacent particles, which results in mild soft agglomeration of m-BTO powder.

BTO and m-BTO have morphological differences, which give intuitive microscopic evidence for successful RL adsorption. The RL organic coating effectively controls the surface energy of BTO particles, which is crucial for the improved surface hydrophobicity and dispersion performance of modified particles.

EDS elemental analysis was done to quantitatively analyze the surface elemental composition changes before and after modification, and the results are shown in Table 3. Based on the data obtained, it can be concluded that the pristine commercial BTO powder contains 24.59 wt% carbonaceous impurities. These impurities were derived from the residual carbonates and organic precursors produced during the powder synthesis process. After using RL modification and ethanol washing, the carbon content of the m-BTO decreases to 11.01 wt%. This value is higher than the expected carbon content of modified BTO with 1 wt% RL, because carbon conductive adhesive was used during SEM observation, and the carbon element in the adhesive may be detected by EDS. This also is the reason of the high carbon content of 24.59%. The mass fractions of the Ba and Ti elements increase from 36.37 wt% and 16.06 wt% to 45.47 wt% and 20.46 wt%, respectively. This variation shows that the ultrasonic dispersion, thermal stirring, and ethanol washing processes effectively removed most of the surface carbonaceous impurities, enabling the purification of BTO particles. This result is consistent with FTIR, XPS, BET, and contact angle test results. It's important to recognize that EDS is a semi-quantitative detection method, which fails to accurately measure the absolute adsorption amount and surface coverage of RL on BTO. Future research will use thermogravimetric analysis to get precise quantitative characterization of RL adsorption capacity.

To further confirm the effect of the modified filler dispersion in polymer matrices, SEM was used to ex-

amine the cross-sectional microstructure of PHBV/BTO and PHBV/m-BTO composites with 12 wt% filler. The results are shown in Fig. 5(c–f). For unmodified PHBV/BTO composites, BTO particle agglomerates were observed in the PHBV matrix, and there were interface defects between fillers and matrix. Regarding the state of the m-BTO powder observed by SEM, the images indicate interparticle adhesion and mild soft agglomeration, which are primarily attributed to the bridging effect of the surfactant molecules. It is crucial to emphasize that this is a physical phenomenon specific to the isolated powder state and does not represent irreversible hard agglomeration. Comparing the PHBV/m-BTO composites, m-BTO particles are uniformly distributed, and there is no large-scale agglomeration. Additionally, the interface between fillers and the matrix is tighter and smoother. The improved dispersion uniformity of m-BTO is due to the RL-induced surface hydrophobic modification, which removes the hydrophilic aggregation tendency of pristine BTO, and enhances interfacial compatibility with the hydrophobic PHBV matrix. This dispersion improvement is in line with the macroscopic performance enhancement of composites.

Mechanical properties of PHBV/BTO composites

The bending strength of pure PHBV, PHBV/BTO, and PHBV/m-BTO composites with different filler loadings are shown Fig. 5(g). The bending strength of pure PHBV is about 43 MPa. For PHBV/BTO composites, the bending strength decreases to 41 MPa at 3 wt% BTO loading, and then increases as increasing filler content, reaching 48 MPa at 12 wt% BTO loading.

For PHBV/m-BTO composites, the bending strength is 45 MPa at 3 wt% m-BTO loading. As the filler content is increasing, the bending strength increases, reaching a maximum of 65 MPa at 12 wt% loading. Compared with unmodified BTO composites at the same loading, the bending strength is improved by 32.7%, and one-way ANOVA confirmed that this performance improvement is statistically significant ($p < 0.05$). The bending strength of composites decreases to 54 MPa at 15 wt% loading, which is due to excessive filler agglomeration and lack of matrix-filler interfacial bonding, resulting increased internal defects of composites and reduced stress transfer efficiency.

The main reason for the improvement in mechanical properties of PHBV/m-BTO composites is the RL surface modification effect. The m-BTO filler is lipophilic, so it has excellent interfacial compatibility with PHBV matrix, which promotes uniform particle dispersion, reduces composite internal defects, and improves interfacial stress transfer efficiency. It is stated that the characterization results of XPS, water contact angle, BET, and SEM can only offer indirect evidence for the improvement of interfacial bonding, not direct proof chemical bond formation. The mechanical

performance enhancement is the whole result of optimizing particle dispersion and improving interfacial compatibility.

The performance of RL-modified BTO was compared with traditional silane-modified BTO, which was reported in the literature. For instance, studies have shown that silane coupling agents-modified bamboo fibers can improve the flexural strength of PHBV or related PHA by approximately 10% to 18% [40]. In this study, RL modification strategy achieved a 32.7% mechanical improvement, which is a competitive modification performance. More importantly, RL modifiers have a unique advantage of full biodegradability and low biological toxicity, which is not comparable to synthetic silane modifiers, and is more suitable for biomedical composite applications. This study lacks parallel comparison experiments for different modifiers under identical experimental conditions, which is a research limitation that needs to be addressed in future work.

The mechanism of RL modification is outlined as follows: RL modification effectively enhances the hydrophobicity and dispersion capabilities of BTO particles. It also improves the interfacial bonding between fillers and the PHBV matrix, and achieves a uniform distribution of fillers. The optimized interfacial structure and dispersion state enhances the stress-bearing and transfer capacity of composites, thus improves the macroscopic mechanical properties.

CONCLUSION

In this study, a green and efficient surface modification strategy was developed, using biological RL as a modifier. The modification mechanism and its effect on the mechanical properties of PHBV/BTO composites were investigated. Experimental results indicate that RL modification enhances the surface lipophilicity of BTO powder, with the water contact angle reaching 125° (optimized m-BTO). XPS and FTIR analyses show that RL molecules are adsorbed stably on BTO surfaces via weak hydrogen bonding and van der Waals forces, not forming strong covalent bonds. BET tests show that RL coating reduces the specific surface area of BTO particles. EDS analysis confirmed that the modification process removed a large fraction of carbonaceous impurities and increased the relative Ba and Ti fractions, while the remaining carbon is attributed to the adsorbed RL layer. TGA further supported these findings by showing a lower mass loss for m-BTO compared to pristine BTO, indicating enhanced thermal stability after surface passivation. SEM observations confirm that RL modification inhibits BTO agglomeration, improving particle dispersion uniformity in the PHBV matrix. Mechanical tests indicate that the optimal 12 wt% m-BTO loading results in PHBV/m-BTO composites having a maximum bending strength of 65 MPa, which is 32.7% higher than that of unmodified PHBV/BTO composites. To the point, RL

can well optimize the surface properties of BTO powder, enable the transition of BTO from hydrophilicity to lipophilicity, and build a stable organic-inorganic hybrid interface. This biodegradable, low-toxicity surface modification strategy has a broad application potential for the production of high-performance biomedical piezoelectric scaffolds and flexible electronic PHBV composites. Meanwhile, there are a few limitations to this study that need to be acknowledged. Firstly, there is no direct quantitative data on RL adsorption capacity and surface coverage on BTO surfaces. Secondly, there are no horizontal comparative experiments with traditional synthetic modifiers under consistent experimental conditions. Thirdly, some characterization results can only provide indirect evidence for interfacial interaction changes. Future research will tackle the above problems, investigate the environmental stability and service durability of RL-modified BTO composites, and check if large-scale industrial preparation is feasible.

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