Film characterization of poly(styrene-butylacrylate-acrylic acid)–silica nanocomposite

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Abstract

To improve the present unsatisfactory UV-shielding and water-resistance properties of many emulsion-type latexes, in this study, poly(styrene-butylacrylate-acrylic acid) (PSBA)-grafted–silica (PSBA-g-silica) hybrid nanoparticles were firstly prepared by suspension–dispersion–polymerization. The resulting hybrid nanoparticles were then used to add into PSBA latex to fabricate PSBA nanocomposites. The influence of PSBA-g-silica hybrid nanoparticles on the interfacial adhesion, film morphology, thermodynamics properties, UV-shielding, water-resistance and mechanical performance of PSBA films were investigated systematically. Our results revealed that filling 1.5 wt% PSBA-g-silica hybrid nanoparticles resulted in significant improvement in interfacial adhesion properties, UV-shielding, water-resistance and mechanical properties. Fabricating the silica-based latex nanocomposite is an effective approach to develop the new emulsion-type paints and adhesives.

Keywords: PSBA latex; Nanocomposite; Water-resistance; UV-resistance; Interfacial properties

1. Introduction

Protecting and preserving the global environment containing the protection of the ozone layer, the prevention against the global-warming, and the recycling of the resources, have become core subjects in the world nowadays. In the field of the architectural paint, the decrease of the amount of volatile organic compounds has strongly been required for a long time [1,2]. This theme has been solved to some degree by using emulsion type paints. However, solvent-type paints are still widely used because the usage of them can make the painting work more efficient than emulsion-type paints as the former dries much faster. The film property of solvent-type paint is generally superior to that of emulsion-type one, which is another reason for the preferable usage of solvent-type paint. Obviously, the technology used for the preparation of emulsion-type paint should be improved to meet the recent requirements either in the performance characteristics or in the environmental field. Many emulsion-type paints have been developed and sold commercially; however, their antipollution and water-resistance properties are far from satisfactory [3].

In recent years, organic–inorganic nanocomposites with well-defined structure and morphology are very interesting and promising class of materials as their potential use in a wide range of conventional application fields [4]. Especially silica-based organic–inorganic hybrid materials can be potentially used in many fields such as plastics, rubbers, coatings, etc. [5–7]. Zhang [8] prepared poly(methacrylic methacrylate) (PMMA)/silica hybrid materials via sol–gel process, which possessed the high transparency and heat-stability. Bokobza et al. [9] reported that silica sol modified by silane coupling agents was mixed with the acrylate monomers to obtain the silica-based hybrid films by ultraviolet radiation. Chang et al. [10] prepared poly(methacrylic methacrylate) (PMMA)/silica hybrid materials via sol–gel process, which possessed the high transparency and heat-stability. Bokobza et al. [9] reported that silica sol modified by silane coupling agents was mixed with the acrylate monomers to obtain the silica-based hybrid films by ultraviolet radiation. Chang et al. [10] prepared PMMA/silica nanocomposites via in situ polycondensation of alkoxysilane in the presence of trialkoxysilylane-functional PMMA. Xia et al. [11] used the ultrasonic induced encapsulating emulsion polymerization technique to prepare polymer/inorganic nanocomposites.

In the polyacrylate/silica nanocomposites, silica (hard fillers) can improve the strength, adhesion, durability, and abrasion
resistance of the polymer materials. The ideal properties of polymer/silica nanocomposites obtained should be dependant strongly on the uniform dispersal of nanosilica in polymer matrix. However, nanosilica, because of a large surface area, is easy to generate silica aggregates embedded in a polymer matrix and resulting in none of the anticipative enhancement in mechanical properties [12]. Therefore, the surface modification of nanoscale fillers becomes necessary in the preparation of nanocomposites. Nanoscale fillers are usually organically modified in order to reduce the filler aggregation and enhance the interaction between the filler and polymer matrix, consequently acquiring a uniform dispersion of the filler in the polymer matrix. Nowadays grafting polymers onto silica surfaces is attracting more and more interest due to their important applications ranging from colloidal stabilizers [13,14] to nanocomposite materials [15,16].

The preparation of organic–inorganic nanohybrids by emulsion polymerization is much preferable because of its easy processability. Percy et al. has been reported the preparation of multi-core-type acrylic/silica nanocomposite particles in an emulsion: the reported particle sizes were 110–220 nm in diameter, while the size of silica core was 13 or 22 nm [17]. Ma et al. has been reported recently that polyacrylate/silica nanocomposite was prepared via sol–gel process by in situ emulsion polymerization [18].

In the present paper, PSBA-g-silica with core–shell microstructure obtained via the dispersion–suspension–polymerization was filled into PSBA latex to prepare PSBA nanocomposite film. The shell of the hybrid nanoparticles was designed to have the same composition as polymeric matrix. This PSBA shell is supposed not only to inhibit the aggregation of nanosilicas but also to bring strong interaction with PSBA matrix and thus resulting in enhanced interfacial adhesion. The effect of PSBA-g-silica hybrids on the properties of PSBA nanocomposite film was studied in detail with the aim to require a broad understanding of the nature of the interaction between hybrid nanoparticles and PSBA matrix. Our results demonstrate the significant improvement in comprehensive properties of PSBA nanocomposites, which is important for the application of silica-plus-latex in paints or adhesives industry.

2. Materials and methods

2.1. Materials

Fumed silica (SiO₂) nanoparticles were supplied by Shanghai Chemical Co., China, with an average diameter of 25 nm. The particles were dried at 120 °C under vacuum for 24 h to eliminate the physically absorbed and weakly chemically absorbed species. Silane coupling agent (γ-methacryloxypropyl trimethoxy silane (MPS)) was provided by Nanjing Shuguang Chemical Industry Co. Ltd., China. Technical-grade monomers n-butyl acrylate (BA), styrene (St), both with 10–20 ppm of hydroquinone monomethyl ether (MEHQ) and acrylic acid (AA) purity of 99% were purchased from by Guangzhou Langri Chemical Co. Ltd., China. BA and St were distilled under vacuum prior to use to remove trace amount of inhibitors. AA was used as received. The emulsifiers (such as nonyl phenol polyoxyethylene ether (OP-10), sodium dodecyl sulfate (SDS)) and potassium persulfate (KPS) were used as received. Doubly deionized water (DDI water) was used throughout the work.

2.2. Preparation of PSBA-g-silica

Functionalized nanosilica with a silane coupling agent (MPS) was carried out as follows: 2.1 mL of MPS was added to a suspension of 4.0 g of nanosilica in toluene solution. After ultrasonic agitation for 30 min, the suspension was refluxed for 10 h under the protection of dry nitrogen flow. Afterwards, the nanosilica was filtrated and extracted with ethanol for 24 h to remove the excess silane absorbed on the particles, and then the silane treated nanosilica was dried in vacuum at 50 °C for 24 h.

The preparation of PSBA-g-silica was performed via suspension–dispersion–polymerization according to our recent work [19] by the following process: 1 g of the MPS-functionalized nanosilica was dispersed in the aqueous solution, in which 2 g of isopropanol, 0.03 g SDS and 0.015 g of OP-10 and 95 ml of DDI water were added. Having experienced ultrasonic agitation for 30 min, the suspension was added into the 250-ml jacketed glass reactor equipped with reflux condenser, thermocouple, stainless-steel stirrer, nitrogen inlet and heated under constant stirring (130 rpm) with protection of nitrogen flow. Once the reaction temperature reached 75 °C, the initiator (KPS), which was dissolved in a definite amount of distilled water, was added into the reactor vessel. Then the monomers 6 g (the weight ratio of styrene, n-butyl acrylate and acrylic acid was 48:48:2) were fed into the reactor in a separate stream with constant flow rates slow enough to reach monomer starved conditions. The time of feeding was 90 min and after addition of the monomers, the system was under reaction for additional 3 h. The obtained products were dried at 45 °C under vacuum after suspension centrifugation and washing process. The hybrid products were extracted with acetone for 24 h in a Soxhlet apparatus to remove the ungrafting polymer and dried to a constant weight under vacuum. The ungrafted PSBA was found approximately 30 (wt)% of PSBA-g-silica.

PSBA latex were prepared by the conventional emulsion copolymerization using the same monomer’s ratios (St:BA:AA 48:48:2) as encapsulation of silica experiments. A detailed recipe was given in Table 1.

Table 1

A typical recipe used for the synthesis of PSBA latex through emulsion polymerization

<table>
<thead>
<tr>
<th>Components</th>
<th>0.108 g</th>
<th>0.015 g</th>
<th>0.03 g</th>
<th>0.05 g</th>
<th>2.1 g</th>
<th>2.1 mL</th>
<th>95 mL</th>
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<tr>
<td>Styrene (12.9 g)</td>
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<td>n-Butyl acrylate (12.9 g)</td>
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<td>Acrylic acid (12.9 g)</td>
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<td>K₂S₂O₃</td>
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<td>SDS (0.5 g)</td>
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<td>OP-10 (0.27 g)</td>
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<td>NaHCO₃ (0.3 g)</td>
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<td>Distilled water (33)</td>
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</table>
2.3. Preparation of PSBA composite film

Stable colloidal suspension was obtained by dispersing PSBA-g-silica into PSBA latex uniformly. A dry film is obtained when the composite suspension was cast in a poly(tetrafluoroethylene) (PTFE) dish and slowly dried in a controlled atmosphere.

2.4. Static immersion test

Static immersion test is considered as a standard method that evaluates water resistance of films using the gravimetric method. Samples of films with a dimensions of 2 cm × 2 cm × 0.15 mm were immersed in distilled water at 25 °C. At specific time intervals, the samples were removed and were blotted with a piece of paper towel to absorb excess water on the surfaces. Weight change was calculated by Eq. (1) and expressed as a function of time.

\[ E_{sw} = \frac{W_c - W_o}{W_o} \times 100\% \]  

(1)

Where \( E_{sw} \) is the water uptake ratio of film, \( W_c \) denotes the weight of the film at different times and \( W_o \) is the dry weight of sample.

2.5. Characterization

Fourier transform infra-red (FTIR) spectrum of PSBA-g-SiO₂ hybrid nanoparticles was recorded on a IFS66/S using KBr pellet. Transmission electron microscope (TEM) micrographs were taken by a TE CHAI-12 (Philips). The samples were diluted up to 20 times and dropped on a copper grid for TEM analysis. Silica or PSBA-g-silica dispersions were prepared in a ultrasound bath with a frequency of 5 kHz. TGA thermograms were recorded on a NETZSCH STA 409PC thermogravimetric analyzer from 25 to 600 °C at a heating rate of 10 °C/min under the flow of anhydrous air. The PSBA-g-silica sample was dried in vacuum at 50 °C for 24 h before TGA measurement. Differential scanning calorimetry (DSC) thermograms were recorded on a 204 F1 from Netzsch Geratebau GmbH Instruments. The samples were weighed on an analytical electrobalance to 9–10 mg for this analysis, the reference used was Al₂O₃ and the cooler used was liquid nitrogen. To eliminate water influence (crystal water), two runs were scanned, from −30 to 70 °C, held at this temperature for 10–15 min and run again from −30 to 70 °C at a heating rate of 10 °C/min under N₂ flow. The morphologies of the fracture surfaces were observed by using a scanning electron microscope (SEM) (HITACHI-S-2150). The specimens were fractured after immersion in liquid nitrogen. The fractured surfaces of specimen were coated with a thin layer (10–20 nm) of gold–palladium before SEM examination [19]. Tensile properties of the sample films were determined with an WDW-5 universal material testing machine at a crosshead speed of 50 mm/min. The sample membrane was dried in vacuum at 50 °C for 24 h to eliminate the effect of water. The membrane was cut into specimen with dimensions: 8 cm × 2.5 cm × 0.15 mm, dog-bone shape. The middle dimensions are: 50 mm × 6 mm × 0.15 mm according to GB 7228-87 of the national standard. All the mechanical tests were carried out at 25 °C. Static contact angles were measured with a FACE contact angle analyzer (First Ten Ångstroms, Virginia, USA) using the sessile drop technique. The measurements were carried out at room temperature in air with deionized water as the probe liquid. 25 µL liquid droplets were deposited onto the sample surface through a gauge 22 sized dispensing needle at a rate of 5 µL/s. Each contact angle reported here is an average of at least five measurements. Atomic force microscopy (AFM) topographs were obtained using a Digital Instruments Multimode IIIa atomic force microscope equipped with an E-scanner. Tapping mode silicon nitride cantilevers TESP with nominal spring constants of 20–100 N/m and nominal resonance frequencies of 200–400 kHz were employed. A piece of freshly cleaved mica (ca. 5 mm × 5 mm) was used as a substrate of film preparation. To minimize possible contamination of the surface by ambient air, each sample was freshly prepared just before the AFM experiments.

3. Results

3.1. Characterization of PSBA-g-silica hybrid nanoparticles

MPS is well accepted as a suitable coupling agent for fabricating polymer-g-silica hybrids [21]. In general, a certain amount of homopolymer is an inevitable product generated during graft polymerization. To separate the ungrafting PBSA, the hybrid particles were extracted with acetone for 24 h. Fig. 1 shows FTIR spectrum of extracted PSBA-g-silica. It can be seen that there is a very pronounced band appearing at 1108 cm⁻¹, together with two less pronounced bands at 806 and 467 cm⁻¹, corresponding to the vibration absorption of Si–O–Si groups. The peaks at 2949, 1736, 1458, 756 and 697 cm⁻¹ are associated with the characteristic vibration of carbohydrate (CH), carbonyl (C=O), methyl (CH₃) and benzyl groups. These results suggest that there are chemical bond between copolymer PSBA and silica.
Fig. 2 illustrates the TGA thermograms of purified PSBA-g-silica. The content of the polymer grafted on the silica can be measured from the residue weight ratio of the hybrid particles. Weight loss for PSBA-g-silica hybrid is determined to be 50.2 wt%, which indicates that the content of PSBA grafted on the silica is near 50 wt% of the hybrid. The grafting efficiency on nanosilica in present condition of suspension–dispersion–polymerization is much higher than previously report where only a small amount of PMMA was grafted onto the talc by the in situ polymerization of MMA on the talc surface [21].

Typical TEM micrographs of initial nanosilica and PSBA-g-silica hybrid are shown in Fig. 3. Fig. 3a shows that the untreated silica has an average diameter of 20–30 nm, and most of the particles show aggregated appearance; Fig. 3b shows the morphology of PSBA-g-silica hybrids, which are regular spherical morphology with diameter ranging from 80 to 90 nm. Moreover, there is no exposed nanosilica observed in the PSBA-g-silica hybrids, suggesting that nanosilica has been encapsulated by the PSBA copolymer effectively. The thickness of grafted polymer is estimated to be 25–35 nm. The TEM micrographs confirm that a thick layer of PSBA has been grafted on the nanosilica surface.

3.2. SEM morphology

Fig. 4 shows the SEM fractographs of the PSBA and its nanocomposite films. It can be seen that the fractographs are strongly dependent on the filling content of PSBA-g-silica hybrids. The fractograph of PSBA filled with 1.5 wt% hybrids demonstrates a layer-like rough appearance (Fig. 4c), indicating an adhesive interface. The edges and corners of the parent PSBA are smoother in comparison with that of PSBA filled with 0.5 wt% hybrids (Figs. 4a and 4b). When further increasing the filling content to 2.0 wt%, the fractograph does not show increased roughness (Fig. 4d) compared with that filled with 1.5 wt% hybrids. This result suggest that PSBA latex filled with 1.5 wt% hybrid nanoparticles led to best interfacial properties.

3.3. Film morphology

PSBA latex and its composite with 1.5 wt% PSBA-g-silica were cast, the resulting films show flat and transparent. The colloidal interactions between the latex particles and the PSBA-g-silica hybrids were further explored using AFM. The diluted PSBA latex and its composite suspension were cast onto freshly-cleaved mica sheets to form thin film. The typical AFM topographic images (4 µm × 4 µm) of PSBA and its nanocomposite films are shown in Fig. 5. The AFM result indicates that the morphology of PSBA composite film is rather smooth. This is supported by the terrace height of around 7.4 ± 0.8 nm along a typical line profile of the AFM image (Fig. 5b) and the low root-mean-square (RMS) roughness of 13.8 ± 1.1 nm. However, on parent PSBA surface, the averaged terrace height and RMS roughness were 10.1 ± 1.0 and 21.1 ± 2.1 nm, respectively (Fig. 5a).
3.4. Glass transition temperature ($T_g$)

Fig. 6 shows DSC curves of PSBA and its nanocomposite film. From Fig. 6, the $T_g$ values for PSBA, PSBA filled with 0.5 and 1.5 wt% hybrids were measured to be 18.8, 20.9 and 23.1°C. It is obvious that $T_g$ increases with hybrid filling, suggesting the hybrids play a role in inhibiting polymer segmental motion. According to Eisenberg’s model, the interaction of polymer chains with nanosilicas reduces the mobility of polymer chains and leads to the formation of immobilized and restricted mobility regions around the filler particles. The DSC spectra clearly indicated the restricted segmental motion and polymer–hybrids interactions. These results are consistent in that of SEM and AFM.

3.5. UV properties

The ultraviolet–visible (UV–vis) absorbance spectra of the PSBA and its nanocomposite films are shown in Fig. 7. From Fig. 7, it can be seen that intensity of absorbance in the wavelength range of 220–270 nm decrease with increasing filling content, which suggests that PSBA composite films have the UV-shielding property. Moreover, there is no absorbance increase in the visible light range, which can prove that the hybrid nanocomposites have not aggregated in the PSBA matrix.

3.6. Water uptake

The water uptake of PSBA and its composite films is shown in Fig. 8. From Fig. 8, it can be observed that the water uptake decreases with increasing filling content. The water uptake of PSBA film was about 25.6%, while the water uptake for PSBA nanocomposite films filled with 0.5, 1.0, 1.5 and 2 wt% hybrids became 13.1, 11.8, 7.0 and 5.4%, respectively. Our results demonstrate obvious water-resistance characteristic of PSBA composite film, which may be due to the formation of cross-linking PSBA structure.

3.7. Wettability measurement

Fig. 9 shows the water contact angle of PSBA and its composite films, reflecting an increase of hydrophobicity with the increase of the filling content. The water contact angle for PSBA film is around 10°, which demonstrates obvious hydrophilic property due to the introduction of surfactants in
the process of emulsion polymerization. However, the water contact angle becomes 52° for PSBA nanocomposite filled with 2.5 wt% hybrids. This enhancement of hydrophobicity is an important factor to lead to a good performance in water-resistance. The increase of water contact angle should be due to the formation of the hydrophobic cross-linking PSBA structure.
3.8. Mechanical properties

Fig. 10 shows the relationship of the mechanical properties and the filling content of PSBA nanocomposites. It can be seen that both of the tensile strength and elongation to break increase with the increase of filling content until 1.5 wt%, followed by a reduction at higher filling content. The PSBA nanocomposite filled with 1.5 wt% hybrids show not only adhesive interface but also maximum value in mechanical properties. Its tensile strength increases form 5.2 to 7.9 MPa, and the elongation to break increases from 262.6 to 414.7% in comparison with that of unfilled PSBA film.

4. Discussion

4.1. Dispersion of PSBA-g-silica hybrid nanoparticles in PSBA matrix

A homogeneous dispersion of nanosilica in the polymeric latex is a difficult task due to strong aggregation tendency of nanoparticles. Nanosilicas have great surface energy, and the dispersion process of nanoparticles in the latex is not spontaneous. Consequently, the anticipative properties such as strength, adhesion, durability, and abrasion resistance of the nanosilica-based-polymeric nanocomposites cannot be fulfilled.

The suspension of PSBA latex filled with PSBA-g-silica was found highly uniform and stable. There is no significant amount of phase separation or sedimentation over a period of weeks. The good suspension stability is believed to be correlated with the designed structure of silica encapsulated with PSBA shell. This polymeric shell effectively inhibits nanosilica agglomeration and provides excellent compatibility with PSBA matrix due to the compatibility of shell of hybrid nanoparticles and matrix. Consequently, the PSBA-g-silica hybrid nanoparticles can be uniformly dispersed into PSBA latex.

The resulting PSBA nanocomposite film from the stable PSBA composite latex shows the smoother morphology of PSBA nanocomposite film in comparison with that of PSBA film (Fig. 5), which suggests that dispersion scale of hybrid nanoparticles in PSBA matrix is within nanoscale. Moreover, in AFM image, the dispersed or the continuous phase cannot be discerned (Fig. 5b). This phenomenon is very interesting and quite different from the other report [22] that PVA-functionalized nanotubes are finely and randomly dispersed in polymer matrix revealed by AFM image. The results are believed to the strong interaction produced between hybrid and PSBA matrix due to the excellent compatibility of PSBA-g-silica and PSBA matrix. The ultraviolet–visible (UV–vis) absorbance spectra demonstrate that there is no absorbance increase in the visible light range (Fig. 7), which suggests that the hybrid nanocomposites have not aggregated in the PSBA matrix.

4.2. Interfacial adhesion

It is well known that the more dispersed the nanosilica is, the better the interface between silica and polymeric matrix it achieves. The surface hydroxyl groups of the untreated silica have an increased tendency to create hydrogen bonds, which directly leads to the formation of aggregates. Moreover, the hydrophobic polymer does not wet or interact with the hydrophilic fillers because of their differences in surface energy [20]. Therefore, the surface modification of nanoscale fillers becomes necessary in the preparation of nanocomposites to improve the dispersion of filler into polymeric matrix and enhance the compatibility between inorganic filler and polymer. In present system, nanosilicas were successfully encapsulated with PSBA copolymer, the aggregation of nanosilica decreases greatly and the interfacial interaction increases due to the compatibility of hybrids and PSBA matrix. Consequently, the good interfacial adhesion achieves (Fig. 4). Only low content of filling (1.5 wt%) is required to obtain excellent interface adhesion (Fig. 4c), which indicates the effectiveness of the PSBA-g-silica hybrid filler.

4.3. Properties of PSBA nanocomposites

The ideal properties of polymer/silica nanocomposites obtained should be dependant strongly on the uniform dispersal of nanosilica in the polymer and the interfacial adhesion. In the present system, when the filling content is less than 1.5 wt%, the interface could transfer stress from matrix to hybrid particles, and improve the yield stress accordingly. However, too high concentration of hybrid nanoparticles filled in PSBA matrix will lead to the aggregates of hybrid nanoparticles in matrix, which would reduce the contact area and create physical defect in composites, all of which will decrease the effective interfacial interaction, and thus the mechanical strength of composites.

One of benefits of emulsion polymerization is the absence of organic solvents. Therefore, emulsion-type paints and adhesives have been widely investigated in research and industry fields [1,2]. However, the antipollution and water-resistance
properties of emulsion-type paints and adhesives are far from satisfactory performance characteristics comparing to that of solvent-type paints [3]. Therefore, the properties of latex have to be improved for developing a new kind of latex, which is applied in paints and adhesives industry. The present PSBA nanocomposite latex has good properties in water-resistance and UV-shielding in addition to the above mechanical properties. Our results demonstrate that fabricating the silica-based latex nanocomposite is an effective approach to develop the new emulsion-type paints and adhesives.

In conclusion, the good dispersion of PSBA-g-silica nanoparticles in PSBA matrix brings in improved interfacial adhesion between inorganic filler and polymeric matrix and thus results in the enhanced properties in mechanical properties, thermo-resistance, water-resistance and UV-resistance properties.

5. Summary

In this study, to inhibit the aggregation of nanosilica and improve the compatibility of fillers with polymeric matrix, PSBA-g-silica hybrid nanoparticles were designed and prepared by suspension−dispersion−polymerization. The resulting hybrid nanocomposites were then added into PSBA latex to fabricate PSBA nanocomposite film. The PSBA nanocomposite films were found to be transparent and smoother morphology in comparison with that of parent PSBA film. The PSBA nanocomposite films have characteristics of UV-shielding, water-resistance and good mechanical properties compared to unfilled PSBA films. 1.5 wt% of these hybrid nanocomposites filled results in good adhesive interface and strong interaction between hybrids and PSBA matrix, suggesting the effectiveness of PSBA-g-silica hybrid nanoparticles as a kind of filler to PSBA matrix. This technology is valuable for the development of new emulsion-type paints or adhesives.

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