Study on the mechanical properties of shear thickening fluid-filled polyurethane foam composites

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Abstract
The previous work on nanocomposites containing silica nanoparticles has indicated that when a relatively high silica nanoparticles content is used, reinforcing effect of that weakens due to the formation of agglomerates of nanoparticles within the polyurethane matrix. Therefore, in this paper, shear thickening fluid-enhanced polyurethane foams were synthesized to resolve such problems. Because hydroxyl groups can form hydrogen bonds with some polar molecules, several layers of molecules in PEG-400 can be adsorbed on the surface of silica nanoparticles. The silica nanoparticles could be dispersed in that polymer matrix very well and surface energy of silica nanoparticles decreases. The comparative study of polyurethane foam (PUF) composites based on shear thickening fluid (STF) had been reported. A wt%-dependent SEM analysis was carried out to understand the effects of STF on the cell morphologies of STF PUF. The linear viscoelastic properties of polymer matrix with STF was characterized by parallel plate rheometer. Finally, the mechanical properties were investigated. The results indicated that when the content of shear thickening fluid is more than 7.5%, the synergistic effect of SiO2 with PEG-400 on enhanced PUF was a major cause of improvement of its mechanical properties.

1. Introduction

Polyurethane foam (PUF) is widely used in insulation, lightweight structure, cushioning materials and other fields because of its moderate mechanical properties and low cost [1–3]. However, the hardness and strength obtained by adjusting crystallinity of hard and soft segments is limited. As a structural material, the mechanical properties of pristine PUF are usually unsatisfactory [4]. In recent years, researchers have studied enhanced PUFs containing titanium dioxide (TiO2) [5], silicon carbide (SiC) [5, 6], graphene oxide (GO) [7], carbon nanotubes (CNTs) [8], silica nanoparticles (SiO2), glass fiber [9], and carbon fabrics [10]. The addition of fabrics and fibers limits the lightweight application of PUF composites [11]. However, silica nanoparticles have been widely used due to their low cost and the potential to greatly improve mechanical properties by introducing small amount of silica nanoparticles into polymer matrix [12]. silica nanoparticles achieves high performance of polyurethane foam from two aspects of resin properties and cellular structure. On the one hand silica nanoparticles has the characteristics of nanomaterials, which enhances the of resin matrix and benefits the stress transfer from the covalent interface formed between the PUF and SiO2. And on the other hand silica nanoparticles can prepare foams with cell smaller and uniform distribution and improve mechanical properties as a heterogeneous nucleation [9, 12]. However, Zhou et al’s studies showed that the hardness and tensile strength of Polyester-based Polyurethane/SiO2 nanocomposite first increased and then decreased as the silica nanoparticles content increased further [13]. A possible mechanism was when a relatively high silica...
nanoparticles content was used, the formation of agglomerates of silica nanoparticles within the polyurethane matrix were favoured by increasing the silica nanoparticles content likely due to stronger hydrogen bond interactions between the silanol groups on the silica nanoparticles [13–15]. Thus, fine dispersion of the silica nanoparticles was considered to be a key factor to obtain optimum properties [16]. In the interest of achieving good dispersion of silica nanoparticles and increasing the interactions between silica nanoparticles and PUF matrix, surface modification of silica nanoparticles was carried out. The most common reagents for that are based on silanes, such as 3-methacryloyloxypropyl trimethoxy silane [17–20]. Luo et al evaluated the effects of the addition of the silica nanoparticles modified with tetraethyl orthosilicate and 3-methacryloyloxypropyl trimethoxy silane on the mechanical properties of PU. The results indicated that the hardness and tensile strength of PU reinforced by modified silica nanoparticles was enhanced remarkably compared with neat PU and that with unmodified silica nanoparticles. That was because that the functional groups C─O on the surface of modified silica integrated with the polymer chains as chemical junction in the network of polymer which could improve the compatibility between nanoparticles with polymer matrix [21]. Nevertheless, the reaction of this kind of modifying agents with silica surface suffers from the possibility of side-reactions that may lead to formation complex surface layers [22, 23]. For these reasons, shear thickening fluid (STF) consisting of SiO2 and PEG-400 was used in PUF as a new kind of reinforcement material in this study. STF made up of dispersed and suspended solid particles in fluids that have a colloidal nature. Under the high-speed shear rate the apparent viscosity of shear thickening fluid increases sharply which shows solid-like form suspension. Then the impact force disappears, it quickly returns to the original flexible state [24]. Since the unique rheological properties and good energy absorption capacity, STF is used far more widely for body armors, reducing vibration and absorbing energy [25]. The general schematic of STF was shown in figure 1. Raghavan’s articles indicated that ethylene glycol was adsorbed on the surface of nanoparticles, forming a solvation layer. Because polyethylene glycol is polar and they preferentially formed hydrogen bonds with the silanol groups on surface of silica nanoparticles. The silica nanoparticles could be dispersed in that polymer matrix very well and surface energy of silica nanoparticles decreases [26–29]. This is a new method to solve the agglomeration of silica nanoparticles.

Li et al proposed STF/PU rigid foam composite sandwich structure. The mechanical properties of PU composites were strengthened. However, the influence of the silica and polyethylene glycol on the properties of composites were analyzed separately in this paper without considering its synergistic effect [30]. Therefore, this design incorporates reticulated foams filled with SiO2/PEG–400 and shear thickening fluid into existing foam and to analyze the enhancement function and mechanism of shear thickening fluid on polyurethane foam [30–32]. To confirm the characteristics of these materials, compression tests, scanning electron microscope (SEM) analyses, plate-on-plate rheometer and FTIR spectrometer were conducted for each PUF type. Their properties were also compared and discussed.

2. Material and experimental

2.1. Materials
Spherical silica (average diameter: 650 nm) purchased from Evonik Degussa Co., Ltd (China) was used as a suspending material. Polyethylene glycol the average molecular is 400 g mol⁻¹ were used as the continuous medium materials supplied by Sinopharm Chemical Reagent Co.Ltd (China). The polyol systems and isocyanates were purchased from the market.

2.2. Synthesis of shear thickening liquid
75 g of SiO2 powder had been added in 25 g of PEG–400 with the stirring speed of 500 r/min. In the preparation process, the stirring process should be continued for a period of time to ensure full dispersion. The prepared samples should be placed in a vacuum drying chamber for 24 h at 25 °C to remove the bubbles.
2.3. Fabrication of enhanced PUFs
Reinforced PUFs were manufactured by a single step method from three constituents, i.e., additives (\(\text{SiO}_2/\text{PEG-400, STF}\)); a polyol system containing catalysts, surfactant and other materials; and an isocyanate system (polymeric methylene diphenyl isocyanate, or MDI). Additives was added in four different amounts, 2.5, 5, 7.5 and 10 wt%, based on the polyol system content. Figure 2 showed the producing process of reinforced polyurethane foam. The polyol system mixed separately with STF were put into the reactor with the rotate speed was set at 1500 r/min for some time. And the isocyanate system also was stirred at a constant rate for a certain period of time. Then, the two reactants were mixed using a homogenizer and poured into an metallic mold to solidify for 15 min Finally, a solid-state polyurethane foam was achieved. In order to further analyze the effect of PEG-400 and SiO\(_2\) in STF on polyurethane foam, the SiO\(_2\)/PEG-400 reinforced PUFs were also made. When the additives were SiO\(_2\) and PEG-400, firstly, silica nanoparticles was added to the polyol system stirring for some time. Then polyethylene glycol was added to the mixture. For the compression test specimen preparation, specimens are fabricated into 50 mm \(\times\) 50 mm \(\times\) 25 mm prisms in accordance with the International Organization for Standardization (ISO 11752:2000).

2.4. Experiment apparatus
In this study, a homogenizer (T50-digital ULTRA-TURRAX, IKA) was used for the homogeneous dispersion of the PUF mixture. The mechanical strength of the specimens was measured on universal testing machine (Instron-3385H, China) under a crosshead speed of 2 mm min \(^{-1}\). Figures 3(a), (b) show a schematic diagram of Instron 3385 H, and photographs of Compressive test. The hardness of the hybrid reinforced PU foam was measured by means of Shore Hardness Tester (LX-A, China) according to GB/T 10807-2006. To explain the effect of shear thickening fluid in polyurethane foam, the FTIR spectra of the samples were carried out in an FTIR spectrometer (Nicolet, USA). The rheological tests were carried out on a parallel plate rheometer (Anton-Paar Physica MCR 301, Austria) in dynamic modes at room temperature. In addition, the cell morphology of the

Figure 2. Preparation process of manufacturing reinforced PUF.

Figure 3. (a) Schematic diagram of Instron-3385H, and (b) photographs of Compressive test.
synthesized SiO₂/PEG-400 reinforced PUF and STF reinforced PUF were observed on a scanning electron microscope (SU1510, HITACHI, Japan).

2.5. Experiment scenario
In order to reveal the effects of the shear thickening fluid on the mechanical characteristics of the PUF, compression testing was conducted until a deformation up to 80% of the initial thickness occurred. In addition, the displacement velocity of 2 mm min⁻¹ was also considered. The compression tests of the reinforced foam was measured with the loading direction of the foam parallel to the foam rise direction. During testing the temperature was maintained at ambient. The entire test was conducted according to International Organization for Standardization (ISO 11752:2000). Table 1 shows the entire test scenario.

3. Results and discussion

3.1. FT-IR spectra
Because different additives have an impact on the structure of PUF, FTIR spectrometer studies were carried out to characterize the synthesized material. Figure 4 shows the FT-IR spectra of both STF, neat-PUF, STF-PUF and SiO₂/PEG-400 reinforced PUF. The obtained spectrum confirms that adding silica nanoparticles and PEG-400 in different form changes the chemical composition of the foam. The characteristic absorption peaks of STF-PUF have no difference of neat PUF, it informs the negligible effect of the STF on the spectra of the PUF. When silica nanoparticles and polyethylene glycol were not added in the form of solution, the structure of composites was affected. The intense bands in the range of 811 cm⁻¹ and 879 cm⁻¹ are attributed to the vibration of Si–O–Si bond and Si–OH bond. The peak at 1643 cm⁻¹ and 1541 cm⁻¹ is related to the vibration of N–H groups. The broad band at 2880 cm⁻¹ is identified as hydrogen vibrating in methylene. The enhanced PUF exhibited three absorption bands which were the same as neat PUF at 3319 cm⁻¹, 1541 cm⁻¹ and 1222 cm⁻¹ could be assigned to stretching vibration of hydrogen bonded N–H, OH groups and C–O–O stretching. It could be clearly seen that the peak at 3455 cm⁻¹ for hydroxyl groups in the STF disappears in the spectra of STF PUF. It was the
evidence of the formation of urethane bonds between the STF and PUF. Raghavan’s articles indicated that ethylene glycol is adsorbed on the surface of silica nanoparticles, forming a solvation layer. As shown in figure 1, because hydroxyl groups could form hydrogen bonds with some polar molecules, several layers of molecules in PEG could be adsorbed on the surface of silica nanoparticles. Accordingly, a small amount of polyethylene glycol reacts in STF with isocyanate due to the formation of solvation layer on the surface of silica nanoparticles [24].

3.2. Morphology of enhanced PUF

Cellular structures are important factors influencing the mechanical properties of polyurethane foams [6, 33]. Usually, nucleating agents are added to reduce the free energy of nucleation to obtain the uniform distribution cells. The amount and dispersion of these nucleation agents influences the nucleation of the PUF [34]. Figure 5 showed a microphotograph and cell distribution of neat PUF STF PUF and SiO2/PEG-400 PUF. As shown in figure 5, with the shear thickening fluid content increasing, cell size decreased with a narrow cell distribution. As previously discussed, a small amount of polyethylene glycol reacts in STF with isocyanate due to the formation of solvation layer on the surface of silica nanoparticles. This reduced the surface energy of silica nanoparticles. The effect of silica nanoparticles as nucleating agent was more significant. When shear thickening fluid content was 10%, the foam cells was relatively smaller and more homogeneous in diameter. The mean cell size of it was 80 μm. The silica nanoparticles acted as herogeneous nucleating agent to reduce the nucleation free energy to produce more samller cells. At the same while, the PEG-400 reacted with isocyanate and affect the foam reaction [8, 32]. With the increasing weight percent of SiO2/PEG-400, the uniformity of cell constantly changes and cell size decreases first and then increased. The mean cell size increased by 27% when the content of SiO2/PEG-400 exceed 5% with a wider cell distribution from 100 μm to 400 μm, which is attributed to the foam reaction affected by PEG-400. With relatively high silica nanoparticles contents, more and more SiO2 could aggregate [13]. All inferences were consistent with the experimental results. Through the comparative analysis, with the increasing weight percent, when SiO2 was adding in forms of shear thickening fluid, the cell structure was more smaller. It could be said that shear thickening fluid acts as the nucleation agent and a proper amount of it is beneficial to cell growth process.

3.3. Hardness

The hardness of polyurethane foam was related to hard segments ratio. The hard segment comprised of diisocyanate and the chain extender. The soft segments consisted of polyether. Figure 6(a) showed the hardness of STF PUF. The data showed that the hardness gradually increased as the STF content increased further. As you can see from the diagram, The hardness for the shear thickening fluid with the weight percent varying form 0 to 10 was 29 Hc, 36.6 Hc, 38.6 Hc, 39 Hc and 50 Hc. When the contents of shear thickening fluid is 10%, the hardness value is the largest. It is increased by 72%. Figure 6(b) showed the hardness of SiO2/PEG-400 PUF. With the mass fraction of silica nanoparticles and polyethylene glycol increasing, the hardness increased first and then decreased. The hardness of SiO2/PEG-400 PUF with the weight percent of silica nanoparticles and polyethylene glycol varying form 0 to 10 was 29 Hc, 45.2 Hc, 31.2 Hc, 22 Hc and 13.6 Hc. When the contents of SiO2/PEG-400 is 5%, the enhanced PUF was the hardest. It is increased by 56%. The hardness of silica nanoparticles was larger than the polymer matrix and the PEG-400 reacted with isocyanate to increase the content of the soft segment. Silica nanoparticles may randomly distribute along the hard segment of polyurea to act as active filler on polyurethane matrix. Therefore, the hardness of reinforced PUF was improved by adding silica nanoparticles. Conversely, the addition of polyethylene glycol reduced the hardness of it [35]. These results could be explained that the reinforcing effects on STF is closely related to its structure. The structure of STF is similar to star-shaped support materials. SiO2 was rich in contents and dispersed uniformly. The hydroxyl groups (−OH) on surface of the solvation layer of SiO2 reacted with isocyanate to form cross-linking point, so the overall hardness increased. Adding SiO2 and PEG-400 separately had no such effect. When the additives were SiO2 and PEG-400, the reaction between PEG-400 and isocyanate plays a major role in hardness reduction. It was another evidence to support that a minimal amount of PEG-400 in shear thickening fluid reacted with isocyanate [1, 9]. It indicated that the most polyethylene glycol in STF binds to silica nanoparticles.

3.4. Rheological behavior of PUF suspension

The cell growth is influenced by the viscoelasticity of polymer matrix. It is vital to verify the viscoelasticity of polymer matrix with different additives. When shear thickening fluid was used as additive, its shear thickening effect could greatly affect the viscoelasticity of matrix. So the viscosity versus shear rate and the storage and loss moduli versus angular frequency of shear thickening fluid was also shown in these figure. As shown in figure 7(a), when the shear rate was greater than 1 s⁻¹, the shear thickening effect appeared, the hydroclusters produced [29]. During this stage, the particle chains gradually extended and the particles contacted with each other. In higher shear rate areas, the chains structure was destroyed, which led to the viscosity decreased [36].
Figure 5. SEM images and cell distribution of PUF composites with varying additives content: (a) neat PUF, (b) 2.5% STF PUF, (c) 5% STF PUF, (d) 7.5% STF PUF, (e) 10% STF PUF, (f) 2.5% SiO$_2$ /PEG-400 PUF, (g) 5% SiO$_2$ /PEG-400 PUF, (h) 7.5% SiO$_2$ /PEG-400 PUF, (i) 10% SiO$_2$ /PEG-400 PUF.
Figure 7(b) showed the linear viscoelastic properties of polymer matrix with different additives to reveal the effect of STF on the foamability of polyol with isocyanate [37, 38]. Overall, as shown in figure 7(b), the polymer matrix with a mass fraction of 10% shear thickening fluid had higher storage and loss moduli. The growth of the cell is divided into two stages: The first is the rapid growth stage, where the film is subjected to strong tensile deformation. Elastic deformation is the main deformation process. The elasticity of blends can inhibit the growth of cells and reduce the size of cells, which is conducive to the formation of more uniform cells. The second is the slow growth stage, where the stress accumulated is relaxed. It is a process of transforming storage modulus into loss modulus. During this process, the strength of the cell wall decreases and damaged cells appear. The blends with higher loss modulus does not resulting in damaged cells [8]. The apparent difference in G' and G'' at low frequencies for the hybrid material is consistent with previous analysis. When the frequency was about 100 s⁻¹, the shear thinning was observed, which made the polymer matrix containing 10% STF viscoelastic gradually weaken [36]. As mentioned earlier, the hydroxyl groups (−OH) on surface of the solvation layer of the SiO₂ reacted with isocyanate to cross-link with PUF. These findings also suggested that the interaction between polymer matrix and shear thickening fluid is stronger. These inferences are consistent with the scanning electron microscope experimental results.

3.5. Mechanical characteristics
The compressive test results of STF PUF and SiO₂/PEG-400 PUF are shown in figures 8 and 9. Effect of STF and SiO₂/PEG-400 loading contents (2.5, 5, 7.5 and 10 wt%) and compression stress of the nanocomposites as shown in figures 8 and 9. In this test, there is no yield point until the sample is deformed by about 10%, and hence the load was read at the point at which a 0.1 mm mm⁻¹ strain occurs [39–41]. Figure 9 showed that the optimum content of adding shear thickening fluid is 10%. When the content of shear thickening fluid is 2.5%,
5% and 10%, the compressive strength is larger than neat foam. When the mass fraction of shear thickening fluid is 10%, compressive strength is increased by 41%. The trend of compressive strength of SiO₂/PEG-400 PUF are not consistent with STF PUF as shown in figure 9. As the mass fraction of silicon dioxide and polyethylene glycol increases, the compressive strength increases first and then decreases.

These results are in good agreement with their SEM images (figure 5) as it was observed [7]. The hardness of silicon dioxide is larger than the polymer matrix. When a relatively high silicon dioxide content was used, nanosilica aggregates, the compressive strength is below that of the neat PUF [13]. And with adding PEG-400, the content of the soft segment increase. It means the strength of SiO₂/PEG-400 PUF reduces. Based on the comparison of test results for different additives, the silicon dioxide and polyethylene glycol separately added into polyurethane foam is different from the state in which ethylene glycol is adsorbed on the surface of nanoparticles, forming a solvation layer. It is noted that the solvation layer avoids the agglomeration of silica compared to that of the SiO₂/PEG-400 PUF. When a relatively high STF content was used, this could result in a significant improvement in mechanical properties. This method greatly improves the agglomeration of silica nanoparticles.

In this work, a possible mechanism for STF PUF as shown in figure 10 [7]. In the STF, several layers of molecules in PEG-400 are adsorbed on the surface of silica nanoparticles, forming a solvation layer. The short-range repulsions caused by solvation layer stabilized the monodisperse particles [24]. The qualitative analysis of the infrared spectra showed that the urethane bonds between the STF and PUF formed. It is not very clear whether the PUF reacted with SiO₂ or PEG-400 adsorbed on the surface of silica. By and large, both reactions ought to proceed. When the additive is STF, the hydroxyl groups (-OH) on surface of the solvation layer of the
SiO₂ reacted with isocyanate to form cross-linking points, so the overall strength increased. Meanwhile, minute quantities of SiO₂ reacted directly cross-linked with isocyanate. When a relatively high silicon dioxide content was used, nanosilica aggregates. And the hydrogen bond in PUF was destroyed due to the reaction of SiO₂ with isocyanate. So the overall strength decreased [33]. As shown in figure 9, the test results indicated that when the content of shear thickening fluid is more than 7.5%, the effect of first reaction dominated. The synergistic effect of SiO₂ with PEG-400 on enhanced PUF was a major cause of improvement of its mechanical properties. When the STF content decreased, the effect of second reaction became dominant. A few SiO₂ nanoparticles may not be enough to show shear thickening effect. The filling of SiO₂ was the main reason for mechanical properties change of STF PUF.

4. Conclusions

In the present study, PUFs reinforced with STF and SiO₂/PEG-400 were synthesized to determine the effects of STF on its mechanical properties and cellular structure. The closed-cell structure of STF PUF was relatively smaller over SiO₂/PEG-400 as shown by the change in cell distribution and mean cell size. When shear thickening fluid content is 10%, the foam cells is relatively smaller and more homogeneous in diameter. The mean cell size of it is 80 μm. The compressive strength of STF PUF was similar to larger than that of neat PUF at ambient temperature, except for at 7.5 wt% STF. The most reinforcing effect appeared for 10 wt% STF. The trend of compressive strength of SiO₂/PEG-400 PUF are not consistent with STF PUF. The mechanical strength were depend on the weight percent of the additives. These results suggest that when the content of additives is over 5%, adding shear thickening fluid to PUF improves the mechanical characteristics over SiO₂/PEG-400-filled PUF. The possible mechanism for STF PUF was proposed. The reinforcing effects on STF is closely related to its structure, which that ethylene glycol is adsorbed on the surface of nanoparticles, forming a solvation layer. It is noted that the solvation layer avoids the agglomeration of silica nanoparticles and reduced surface energy of it. It is a novel method for preparing reinforced PUFs.

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