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Mechanical and thermal properties of polypropylene/modified basalt fabric composites

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ABSTRACT: Basalt fabric (BF) was first treated with silane coupling agent KH550, modified basalt fabric (MBF) was obtained. Then MBF were molded with polypropylene (PP) matrix, and polypropylene/modified basalt fabrics (PP/MBF) composites were obtained. The influence of concentration and treating time of KH550 on MBF were characterized by hydrophilicity and lipophilicity. The tensile strength and morphology of basalt fabric were tested by single filament strength tester and scanning electron microscopy. The mechanical properties of composites were measured with electronic universal testing machine and impact testing machine, and the thermal properties were tested by thermogravimetric analysis and dynamic mechanical analysis. The results showed that the lipophilicity of MBF is improved significantly by KH550 while the tensile is nearly damaged. The mechanical properties of composites are larger than that of pure PP, among which the impact property was improved the most, showing 194.12% enhancement. The thermal stability and dynamic viscoelasticity were better than pure PP; furthermore, the concentration of KH550 virtually had no effect on the thermal stability. © 2015 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2015**, *132*, 42504.

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INTRODUCTION

Basalt fiber is a newcomer to fiber-reinforced polymers and structural composites. It has a similar chemical composition as glass fiber but has better strength characteristics and higher elastic modulus characteristics.^{1,2} In general, basalt fiber has good acid and alkali resistance, weather resistance, excellent heat resistance, sound and heat insulation properties.^{3–7} Compared with Kevlar fiber and carbon fiber, basalt fiber has the features of wider application temperature range of -269 to $+650^{\circ}$ C, higher oxidation resistance, higher radiation resistance, higher compression strength, higher shear strength, and lower cost. Due to these excellent properties, basalt fiber can be widely used in various industrial fields, such as civil application, military affairs, national defense, and other fields.⁷

Basalt fiber and its fabric-reinforced polymer have significant performance enhancement than the traditional resins. Researches about basalt fiber and its fabric-reinforced polymers have gained great achievements. The researchers have used epoxy resin,^{8,9} phenolic resin,¹⁰ polyamide,^{11,12} vinylester, poly-propylene (PP),^{13,14} and other materials as matrix to prepare a series of basalt composites. The studies indicate that basalt fiber

composites not only inherit the excellent physical and mechanical properties of basalt fiber but also maintain the characteristics of the matrix. For example, Wei¹⁵ studied the epoxy resin/ basalt fiber composites and the basalt fibers were modified by hybrid sizing. The results showed that increased tensile strength of basalt fiber and the interlaminar shear strength of the composites was improved. Lu¹⁶ studied the basalt fiber/epoxy resin composites. The comparison of basalt fiber and glass fibersreinforced epoxy resin showed that the mechanical properties and temperature properties of basalt fiber were excellent; basalt fiber still has 67.9% of the Powerful at 500°C. Manikandan¹⁷ studied mechanical properties of basalt fiber and glass fiberreinforced unsaturated polyester composites both with and without acid and alkali treatments of the fabrics. The study showed that the reinforcement of basalt fiber composites is superior to glass fiber-reinforced composites.

As we all know, PP materials have low stiffness, poor wear resistance, poor weather resistance, high molding shrinkage rate, easy oxidization, and other disadvantages. In addition, PP plastic products are very sensitive to the gap effect, especially show poor impact performance at low temperature (below 0° C). In this article, PP was chosen as matrix materials and basalt fabric

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Figure 1. The modifying mechanism of BF with KH550.

as reinforcement, and then the BF/PP composites are obtained. Mechanical properties research shows that the addition of BF significantly improved the mechanical properties of the matrix, and KH550 effectively improved the interface of the composite materials.

EXPERIMENTAL

Experiment Materials

Polypropylene, T30S, was supplied by China Petroleum Dalian Petrochemical Company. Basalt fiber (diameter is 7 μ m, total denier is 800 Tex/bundle) and its fabric (warp and weft density were 10 F/10 mm) was supplied by Russian Gold Basalt Fiber Ltd. 3-Aminopropyltriethoxysilane (KH550) was supplied by Jiangsu Chenguang Coupling Agent Ltd.

Methods

Basalt Fiber Surface Treatment. This experiment used KH550 to treat the surface of BF to improve the surface energy, roughness, and interface adhesion; the mechanism¹⁸ is shown in Figure 1.

Specific operations: A certain amount of basalt fibers were weighed and soaked with ether in order to remove the oil on the surface and dried. Then the fibers were treated with 4%, 6%, and 8% KH550 solution for 0.5–2.0 h at 80°C. Then the fibers were dried under vacuum at 120°C for 2 h.

Preparation of BF/PP Composites. A certain amount of PP resin was weighed and mixed in two-roll mixer, then molded on flat vulcanizing machine. Process parameters: temperatures of the three mold boards were 190, 190, and 185°C, respectively; pressure was 2.5 MPa, vulcanization time was 5 min. A number of PP slices were prepared first by this method, then basalt fabrics were sandwiched between two PP slices, and then molded on flat vulcanizing, finally BF/PP composites were obtained. Pure PP notes for 1#, unmodified BF for 2#, 4% KH550 modified for 3#, 6%KH550 modified for 4#, 8%KH550 modified for 5#. The fabrics in all of the samples was 13.2 wt %.

Test and Characterization

Property Tests of Modified and Unmodified BF. Hydrophilic and lipophilic test of BF. Modified and unmodified basalt fibers were soaked in deionized water and liquid paraffin for 4 h, then centrifuged in ion settler for 10 min, weighed and marked as wet weight: m_1 , then dried in 120°C vacuum oven until reached constant weight; weighed and marked as dry weight: m_2 , at last calculated the fiber water retention rate and oil retention rate by the following formula:

$$W = (m_1 - m_2)/m_2 \times 100\% \tag{1}$$

Mechanics performance test of BF. The mechanical properties of modified and unmodified BF were tested by a monofilament strength tester (Laizhou City Electronic Instrument Limited Company). The experimental conditions were shown as follows: temperature of 20°C, humidity of 65%, the tensile speed of 25 mm/min, and chuck gauge of 5 mm.

SEM of BF. The fiber surface was observed by JSM-6360LV scanning electron microscope (SEM) (Japanese Electronics). In order to prevent charge accumulation, all samples are required to metal spraying process. Experimental conditions: the discharge voltage of 10 kV, the magnification of 100 times and 1000 times.

Property Tests of PP/BF Composites. *Mechanics performance test.* The prepared composites were placed for 24 h; dumbbell-shaped tensile, impact, and bending samples were made with a universal cutting machine. Tensile and flexural tests were oper-ated by the electronic universal testing machine according to GB/T1040-92 and GB/T9341-2000 standards, stretching speed was 50 mm/min, bending test speed was 2 mm/min, and temperatures were both at room temperature. Notched impact strength was tested by UJ-40 impact testing machine according to GB/T1843–1996 standard, at room temperature.

Thermogravimetric analysis (TGA) test. Thermal property tests were performed on 10 mg powdered samples, heating from 0 to 700°C at10°C/min, under nitrogen atmosphere with a TA instrument thermo balance TGA Q50, USA. $T_{5\%}$, $T_{50\%}$, and T_{max} are the temperatures of 5% weight loss, 50% weight loss, and maximum rate of weight loss, respectively.

DMA test. Dynamic mechanical analysis (DMA) tests were performed with a DMA instrument Q80 by TA, USA. The sample was $40 \times 10 \times 5$ mm, the scanning temperature was from 0 to





Figure 2. Processing time of 4% KH550 relationship with water and oil retention rate of BF.

160°C, with a heating rate of 3°C/min, frequency was 1 Hz, and strain amplitude was 20 μ m.

SEM test. The section of BF/PP composites was observed by JSM-6360LV SEM (Japanese Electronics). In order to prevent charge accumulation, all samples are required to metal spraying process. The experimental conditions: the discharge voltage of 10 kV, the magnification of 100 times and 1000 times.

RESULTS AND DISCUSSION

Properties of Modified and Unmodified BF

Analysis of the Hydrophilic and Lipophilic. As shown in Figure 2, when BF were all treated with 4% KH550, the processing time had a greater impact on the hydrophilicity and lipophilicity of fibers. Hydrophilicity of fibers first dramatically reduced, then increased, and finally stabilized with the processing time increased; lipophilicity first increased and then decreased with the processing time increased, because this modification process made the hydroxyl reduced and alkyl increased. After processing for 1.5 h, hydrophilicity increased slightly, but lipophilicity drastically reduced. This is because the processing time was too long and caused the hydrolysis for part of KH550, although the hydroxyl content still increased, the reaction probability of coupling agent and BF greatly reduced, produced alkyl's content greatly reduced, and therefore lipophilicity dramatically declined.

As it also can be seen from Figure 3, the water retention rate of basalt fiber reduced with the mass fraction of KH550 increased, when the process time was 1.5 h; hydrophilicity declined, oil retention rate increased, and lipophilicity improved. From the reaction shown in Figure 1, the hydroxyl on the fiber surface reduced, resulting in hydrophilicity declined; in meantime, the organic groups on the fiber surface formed, which made the lipophilicity improved. Moreover, the higher the concentration of KH550, the more hydroxyl consumed, the more organic groups formed, the more hydrophilic declined, and the more lipophilicity increased, until reached the equilibrium value.

Analysis of Mechanical Properties. It can be seen from Table I that the mechanical properties of modified basalt fibers had



Figure 3. Mass fraction of KH550 relationship with water and oil retention rate of BF, when processing time was 1.5 h.

changed a bit compared with the original, which shows that KH550 would not cause significant damage on the mechanical properties of fibers, and can increase the mechanical properties of fibers in a certain circumstance appropriate concentration of KH550. This is because it can form a more uniform monolayer film on the fibers surface with the appropriate concentration of KH550, which reduced the flaws on the fibers surface, played a reinforcing role to some extent.

Combining the results of tensile properties with the previous hydrophilicity and lipophilicity of fibers, we can know that basalt fibers treated with KH550 not only can achieve the purpose of the surface modification which improved the adhesion between BF and PP matrix, but also will not cause significant damage to the tensile strength. What is more, it can also cover some of the flaws brought by fiber production process to a certain extent.

Analysis of SEM. Figure 4 is the SEM images of the surface of BF in the blending section. Form Figure 4, we can see that the surface of untreated BF is smooth, only a small PP resin punctate distribution attached to the surface, indicating that the adhesion property between BF and PP is poor, while Figure 4(b–d) compared to (a), the surface of BF had a lot of PP resin, which showed that KH550 improved the adhesion properties of BF/PP. Form the graph, it also can be found that the surface of BF treated by 6% KH550 were significantly adhered more PP than Figure 4(b,d), showed that the adhesion of PP/BF are the best when the KH550 concentration is 6%, while the BF surface

Table I. Mechanical Properties of BF Treated with Different Mass Fraction of KH550 (Process for 2 h)

Fibers	Tensile strength at break (GPa)	Elongation at break (%)
Untreated BF	7.5	3.9
4% KH550-treated BF	7.3	4.7
6% KH550-treated BF	7.5	4.4
8% KH550-treated BF	7.3	8.1





Figure 4. The SEM images of BF: (a) untreated, (b) treated by 4% KH550, (c) treated by 6% KH550, and (d) treated by 8% KH550.

treated by 8% KH550 occurred agglomeration and dispersed unevenly, resulting in binding capacity between BF and PP becomes weak, which is consistent with the results of mechanical properties test previously mentioned.

Properties of the BF/PP Composites

Analysis of Mechanical Properties. In BF/PP composite materials, basalt fabric are the main bearers of loads in composites as the reinforcement. The fracture strength of basalt fabric played a decisive role on the mechanical properties of composites.

Figure 5 showed that the breaking strength of PP/BF was all increased except unmodified PP/BF which decreased slightly compared with pure PP. Because basalt fabric are inorganic materials whose surface is inert to the organic compounds, leading to poor adhesion of interface between BF and PP matrix, which is easy to divorce and to form stress concentration that reduces the strength of composites. The addition of KH550 improved the interface of the combination force and between BF and PP matrix, so as to enhance the tensile properties, which explained the breaking strength of 4%, 6%, and 8% KH550-modified BF/PP were all higher than pure PP. Different KH550 concentration led to different tensile strength, of which 6% was the best, increasing by 85.6%. Combined with the previous results of monofilament lipophilicity, we can see that in this condition the KH550 concentration was higher, and groups on the BF surface reacted sufficiently and formed the most organic groups and lipophilicity was the best, the combination force between BF and PP matrix was also the strongest, BF can share most of the tensile loads, therefore tensile strength was

the highest. The combination force between BF and PP matrix was weaker and the interface layer formed was weaker when the KH550 concentration was too low or too high, therefore, it is easy to rupture when stretching.

As also can be seen from Figure 5, the impact strength of the composites gained a larger increase than pure PP, unmodified BF/PP, 4%, 6%, and 8% KH550-modified BF/PP increased 173.9%, 176.5%, 194.1%, and 160.1%, respectively. When composite materials received impact load, the force first reached the surface layer of the matrix formed damage, and then the load continued and extended to the inner, resulting in damage of the interface between matrix and fabric and squeezed the fabric; when the fabric was damaged, impact load spread out and affected on the third layer of the matrix, which damaged the matrix and fractured the composites. So the impact strength of the material reinforced with fibers were higher than pure PP, basalt fibers modified with 6% KH550 had the strongest combination force with the matrix; therefore, 6% KH550-modified BF/PP materials had the highest impact strength. From the data we can know that excessive organic treatment will lead to the decline of impact strength increase rate. It is probably because the excessive coupling agent treat fabrics would cover too much coupling agent molecules and form partial adsorption of double molecular and multi molecular. Coupling molecules closest to the PP interact with them. Yet other coupling agents around gathered in PP and formed a lot of physical adsorption layer with loose structure, which results in weak phase at the interface of fabrics and PP matrix. Meanwhile, excessive coupling agent can cause condensation and the coupling agent could not fully



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Figure 5. Mechanical properties of BF/PP composites: tensile strength, impact strength, and bending strength.

coated fabrics surface. So it leads to the uneven interface bond strength of fabrics and PP matrix. As the materials withstanding high stress, it will generate the amount of cracks. However, the cracks do not expand along the interface, but forming concentration of stress. So the impact strength of composite materials is decreased significantly. Therefore, the amount of coupling agent should be controlled.

The addition of Basalt fibers can improve the flexural strength of the composites, and with the increase of KH550 concentrations, the bending strength first increased and then decreased. When the concentration was 6%, bending strength reached the maximum value of 7.6%, as shown in Figure 5. This is due to basalt fibers with high modulus and rigidity, good bending performance, so they were able to give the composites a higher bending strength. In addition, the cracks appeared in bending test were from the bottom of the sample, and with the increase of force, the cracks gradually extended along the cross-section of the specimen. To break the specimen, the force needs to destroy not only the PP matrix but also the interfacial layer between the BF and PP. And from the above analysis, we can see that material interface layers were the strongest when modified by 6% KH550; therefore, at this time the bending performance reached the maximum.

Analysis of TGA. From Figure 6(a), we can found that a shift occurred to the direction of higher temperature and lower residual weight for the 6% and 8% KH550-modified BF/PP composites' curves relative to the pure PP; while in Figure 6(b), untreated BF/PP, 6%, and 8% KH550-modified BF/PP composites curves shifted to the right. The thermal degradation rates of PP were low before 400°C; the degradation rates of PP were high and most of the degradation occurred at this stage between 400 and 500°C; and after 500°C, the degradation rate of PP became low again until completely degraded. It revealed that the thermal stability of BF/PP composites had a great increase, which indicated that the BF was able to inhibit the thermal degradation of PP. This is because BF had excellent heat insulation property, the existence of basalt fiber could reduce the heat transfer rate when the composite materials were heated, resulting in the decline for the thermal degradation rate of PP matrix; therefore, the basalt-fibers-reinforced PP composite materials



Figure 6. Thermograms of PP and BF/PP composites under nitrogen: (a) TG curves and (b) DTG curves.

were more stable than pure PP material at high temperature. The 6% and 8% KH550-modified BF/PP composites curves were very close, and the trends of the two cures were also consistent, indicating the concentration of KH550 had little effect on the degradation of PP.

Table II showed that $T_{5\%}$, $T_{50\%}$, and T_{max} of composite materials are improved to some extent compared with pure PP, but the concentration of KH550 virtually had no effect on the temperature for weight loss of composite materials. Specifically, $T_{5\%}$ of unmodified BF/PP, 6%, and 8% KH550-modified BF/PP composites increased by 24, 33, and 29°C; $T_{50\%}$ increased by 33, 33, and 34°C; and T_{max} increased by 20, 20, and 21°C. It indicated that the introduced basalt fibers can effectively inhibit the degradation of PP and improve the thermal stability of PP.

Analysis of DMA. Figure 7 shows the curves obtained from DMA tests of composites, which were treated with different concentrations of KH550. According to Figure 7(a), we can see five curves of storage modulus E' that decreased with increasing temperature, before 80°C, the decline rate was faster. At the glassy state, the polymer molecular chain segments were at the



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Sample	T _{5%}	T _{50%}	T_{\max}
Pure PP	364.5	419	465
Untreated BF/PP	388.5	452	485
4% KH550-treated BF/PP	394.6	452	485
6% KH550-treated BF/PP	397.5	452	485
8% KH550-treated BF/PP	393.5	453	486

Table II. Thermal Performance of BF/PP Composites

Table III. Glass Transition Temperature of BF/PP Composite Materials

Sample	Glass transition temperature (T _g)
Pure PP	83.5
Untreated BF/PP	81.2
4% KH550-treated BF/PP	84.5
6% KH550-treated BF/PP	89.5
8% KH550-treated BF/PP	87.3

frozen state, the conformation of macromolecular chains could not rearrange, so the stiffness of materials was high and the modulus was high as well; with the increase of temperature, molecular chain segments began to move gradually, materials transformed from the glassy state into viscoelastic state, and then modulus was decreased significantly; finally, temperature was so high that the entire molecular chains can move, macromolecular chains showed conformational rearrangement, resulting in decreased modulus continuously, but the decline rate was slower. From the figure, it can be seen that PP composites reinforced by the modified BF showed increased storage modulus, compared to the pure PP, but the storage modulus of unmodi-



Figure 7. DMA curves of composites treated with different concentrations of KH550 (*E*': storage modulus, tan δ : loss factor): (a) *E*' curves and (b) tan δ curves.

fied BF-reinforced PP composites (unmodified BF/PP composites) reduced because the interface performance between the modified fibers and matrix was better, meaning the addition of fibers can increase the rigidity of the material and storage modulus, the situation without modification was reversed.

It can be seen from Figure 7(b) that tan δ values increased as the temperature rose first, reached the maximum at about 100°C, and then declined, but finally continued to increase, and there were very little difference among pure PP, 4%, 6%, and 8% KH550-modified/BF composites, while the tan δ value of unmodified PP/ BF composites was much higher than the others'. Because the peak height of tan δ was in close relationship with the mobility of polymer molecular chain segments and side groups, it correlates with the number and strength of primary or secondary bonding established between the BF and the PP matrix. The bonding was very poor for unmodified BF/PP composite, which was reinforced by BF without KH550 modification.

From Table III, it can be found that the addition of modified basalt fibers can improve the glass transition temperature (T_g) of the composites, and with the increase of KH550 concentrations, the T_g first increased and then decreased. The addition of KH550 improved the combination force of interface between BF and PP matrix. Considering that the glass transition is related with the mobility of chain segments in the amorphous regions, here these segments adjacent to the BF have lower mobility. It is mainly because that the BF hinders movement of PP molecular, thereby leading to an increase in $T_{\rm g}$. However, excessive organic treatment will lead to a decrease in $T_{\rm g}$ of composites. It is due to coupling bilayer adsorption or aggregation structure forming on the surface of basalt fabrics, decreasing interfacial interaction between BF and PP matrix. It can be concluded that adding proper content of coupling agent treatment in BF not only increase the strength of the composite materials but also improve high temperature performance.

Analysis of the Fracture Surface. Figure 8 shows the SEM images corresponding to BF/PP composites with different content of coupling agent treatment BF. Form Figure 8(a), we can see that the surface of untreated BF is smooth. And there are obvious gaps between BF and PP, indicating that the compatibility is poor between untreated BF and PP, resulting in interfacial binding capacity between BF and PP becomes weak. In addition, PP resin is not dispersed into the BF, only a small amount of debonding resin scattered on the surface of BF. Since load cannot be transferred by BF in composites, the composites



Figure 8. Fracture surface image of BF/PP composites: (a) untreated, BF/PP, (b) treated by 4% KH550, BF/PP, (c) treated by 6% KH550, BF/PP, and (d) treated by 8% KH550, BF/PP.

is damaged mainly in the interfacial area. Figure 8(b–d) shows that the interface fuzzy between modified BF and PP matrix, and more PP resin dispersed into the BF. This indicates that the BF with coupling agent treatment can improve the compatibility with PP matrix, resulting in interfacial binding capacity between BF and PP becomes stronger. Under external force, the interfacial layer played a role in transfer stress, BF undertook most of the loads, and played a role in reinforced composites.

CONCLUSIONS

It has been shown that silane coupling agent KH550 was applicable for BF, and BF were modified with KH550, the lipophilicity of BF was improved. In the results, the BF treated by 6% KH550 for 1.5 h resulted in the best lipophilicity. The modification process had little effect on the tensile properties of fibers. The addition of fibers can effectively improve the mechanical properties of the matrix. In terms of the tensile, impact resistance, and bending properties, the properties of fibers modified with 6% KH550 were optimal, which compared with pure PP, respectively, increased 85.6, 194.1, and 7.6%. The thermal stability of BF/PP composite materials was better than pure PP material; in addition, the concentration of KH550 virtually had no effect on the thermal stability. The modified BF/PP composite materials demonstrated higher storage modulus and lower loss factor, which means modified composites have better dynamic viscoelasticity.

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