



Preparation and Property Evaluation of Polypropylene Composites Reinforced by γ -Aminopropyltriethoxysilane-Modified Talc

Dinghao Wang^{1,*}, Andrii Bieliatynskiy¹, Guangtao Zhang¹ and Yajian Deng¹

¹ North Minzu University School of Civil Engineering, Yinchuan, 750021, Ningxia, China

SUMMARY: *In order to improve the mechanical properties of polypropylene, this paper uses flake talc (TA) filled polypropylene (PP) to enhance the mechanical properties of polypropylene (PP) to improve the applicability of polypropylene. However, when unmodified TA is filled with polypropylene, the surface of unmodified TA is hydrophilic and highly polarized. While the polypropylene (PP) matrix is non-polar and hydrophobic, the difference in surface energy between the two makes the inorganic filler in the polypropylene (PP) matrix prone to agglomeration, weak interfacial bonding, and prone to stress concentration, which will reduce the tensile properties of the material while enhancing the rigidity of the composite. In order to improve the interfacial compatibility and load transfer efficiency, the silane coupling agent γ -aminopropyltriethoxysilane (KH550) was used to modify talc (TA). KH550 was hydrolyzed to generate silanol and condensed with the hydroxyl group on the surface of talc (TA) to form a -Si-O-bond, and at the same time, its organic end-groups enhanced the wettability and interactions in the interfacial area, thus inhibiting the agglomeration and enhancing the stress transfer. The modified talc (TA) and polypropylene (PP) were mixed homogeneously and then extruded into PP/TA composites using twin-screw melt extrusion pelletizing and then extrusion molding. The results showed that the tensile strength of the composite material was the best when the talc (TA) content was 10% and the silane coupling agent γ -aminopropyltriethoxysilane (KH550) concentration was 2%, and the tensile strength reached 32.83 MPa (149% higher than that of pure PP). Talc (TA) content of 20%, silane coupling agent γ -aminopropyltriethoxysilane (KH550) concentration of 2% of the composites of the flexural strength of the most excellent flexural strength reached 48.32 MPa (compared with the pure PP to enhance the 127%), in this paper, by varying the content of talc (TA) and the concentration of silane coupling agent γ -aminopropyltriethoxysilane (KH550) to achieve the inorganic filler from agglomeration to dispersion in polypropylene (PP) matrix. Ideas are provided for the design of polypropylene (PP) composites with excellent performance.*

KEYWORDS: *polypropylene; talc; surface modifier; system enhancement; interfacial modification*

1 Introduction.

Polypropylene (PP), as one of the most widely used engineering plastics with the largest production volume in the world, has the advantages of low density, good chemical resistance, good heat resistance, excellent electrical insulation, good processing performance, relatively low price and recyclability, and is widely used in packaging, automotive, construction and medical fields, etc. The relative lack of stiffness, strength, and thermal deformation resistance

*18035628811@163.com

<https://doi.org/10.65102/is20261269>

of PP limits its application in the load-bearing structure and dimensional stability of demanding scenarios. The relative lack of stiffness, strength and thermal deformation resistance of PP limits its application in scenarios with high demands on load-bearing structure and dimensional stability. The introduction of high modulus inorganic fillers to improve the rigidity and dimensional stability of materials is one of the effective ways to enhance the engineering performance of PP.

Talc (TA) is a layered silicate mineral with typical lamellar morphology and high aspect ratio. After twin-screw melt extrusion, the TA lamellae have delamination and orientation potentials, which can form an effective load-bearing phase in the composites, thus enhancing the system rigidity, creep resistance and high-temperature dimensional stability. [1, 2] However, the presence of hydroxyl groups on the TA surface presents hydrophilicity, and the PP matrix is a nonpolar hydrophobic polymer, which leads to the thermodynamic incompatibility of the two: fillers are prone to agglomeration, weak interfacial adhesion, and impeded load transfer; filler particles are more prone to become stress concentrations under external loading, which ultimately weakens the comprehensive mechanical properties of the composites. The key to improve the efficiency of PP/TA composites is to regulate the TA/PP interface and build a stable transition interface layer.

Silane coupling agent can realize inorganic and organic two-phase bonding through inorganic end bonding + organic end compatibility. KH550 is widely used in the surface modification of mineral fillers; it is easy to hydrolyze to form silanol in hydroalcoholic system and condense with the hydroxyl group on the surface of filler to form stable -Si-O- bond; its terminal amino structure can improve the degree of filler surface organization and wettability, and it may introduce polar interactions in the interfacial region, thus enhancing the interfacial shear strength and stress transfer efficiency. [3, 4] Based on this, this paper adopts KH550 to modify the surface of TA, and systematically investigates the effects of TA content and KH550 concentration on the microscopic morphology and tensile and flexural properties of PP/TA composites, and elucidates the mechanism of interfacial modification by combining with FTIR, XRD and SEM characterizations. [5]

2 Experimental part

2.1 Main raw materials

Polypropylene (PP), Nordic Chemical RB206MO; Talc (TA) Table 1 Talc composition (TA), 1250 mesh, Lianfeng Technology Factory Mineral Powder Processing Company; Anhydrous ethanol, analytically pure, Foshan Shilong Chemical Co. Ltd.; γ -aminopropyltriethoxysilane (KH550), Dongguan Kangjin New Material Technology Co.

Table 1: Talcum Powder Composition Table (Unit: %)

SiO ₂	CaO	MgO	Al ₂ O ₃	Fe ₂ O ₃	FeO	F	BaO	K ₂ O	S	Na ₂ O
39.24	29.32	27.14	2.17	0.62	2.36	0.521	0.376	0.326	0.064	0.059

2.2 Instruments and equipment

High-speed disperser; Oven; Microcomputer gantry tensile tester, KY-D4105, Shanghai Horiyang Precision Measuring Instrument Company Limited; Desktop vertical injection molding machine, Z-0020g, RCZJ Company, China; Fourier Transform Infrared Spectrometer (FTIR), ScientificNicoletis20, ThermoFisher Company, USA; X-ray Diffraction (XRD),

D8Advance, Bruker, Germany; scanning electron microscope (SEM), SIGMA-360, ZEISS, Germany; integrated thermal analyzer, STA200, HITACHI, Japan.

2.3 Sample preparation

2.3.1 TA surface modification

The unmodified talcum powder was recorded as UMTA, UMTA was placed in a preheated oven at 80 °C and dried for 2h, and the KH550 modifier solution with a concentration of 1%, 2% and 3% was proportionally configured (KH5500% represents the unmodified test control group), and was heated to 60 °C and stirred magnetically for 30min after the reaction was completed, and then cleaned and filtered by ethanol and acetone and deionized water, and then the treated powder was The treated powder was dried in an oven for 12 h. The modified talcum powder was finally obtained, which was noted as MTA. Fig. 1 shows the flow chart of TA surface modification.

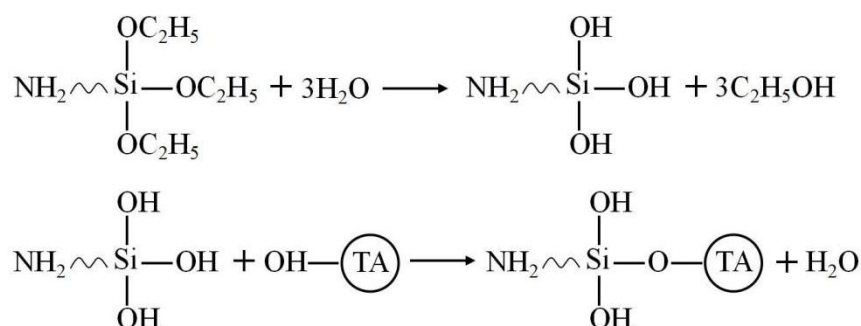


Figure 1: Schematic Diagram of the Mechanism of KH550 Modified Talc (TA)

2.3.2 Preparation of UMTA/PP and MTA/PP composites

Firstly, PP was dried in an oven at 80°C for 2h, PP and UMTA/MTA were put into a high-speed mixer at 2000r for ten minutes, and the obtained materials were put into a twin-screw extruder for extrusion, with the temperature of the heating section at 180-190°C, the speed of the mainframe screw at 30r/min, and the speed of the charging material at 25r/min. Table 2 shows the formulations of the composites. According to the formula in Table X, the mixture of silane coupling agent modified TP and PP is slowly put into the feed port, and the raw material is extruded from the glassy state to the viscous flow state with the head through the melt blending method, and it enters into the pelletizer to cut it into the fine particles of 3-5mm after being manually hauled to the liquid-cooled circulating device. The pellets are evenly put into the desktop injection molding machine 230°C, 27MPa to make standard tensile and flexural test samples. Figure 2 shows the flow chart for the preparation of PP/UMTA and PP/MTA composites.

Table 2: Composite Material PP/TA Formulation

	PP (wt%)	UMTA (wt%)	MTA KH550 1% (wt%)	MTA KH550 2% (wt%)	MTAKH550 3% (wt%)
1	95	5			
2	90% (wt%)	10			
3	85	15			
4	80	20			
5	95		5		
6	90		10		
7	85		15		
8	80		20		
9	95			5	
10	90			10	
11	85			15	
12	80			20	
13	95				5
14	90				10
15	85				15
16	80				20

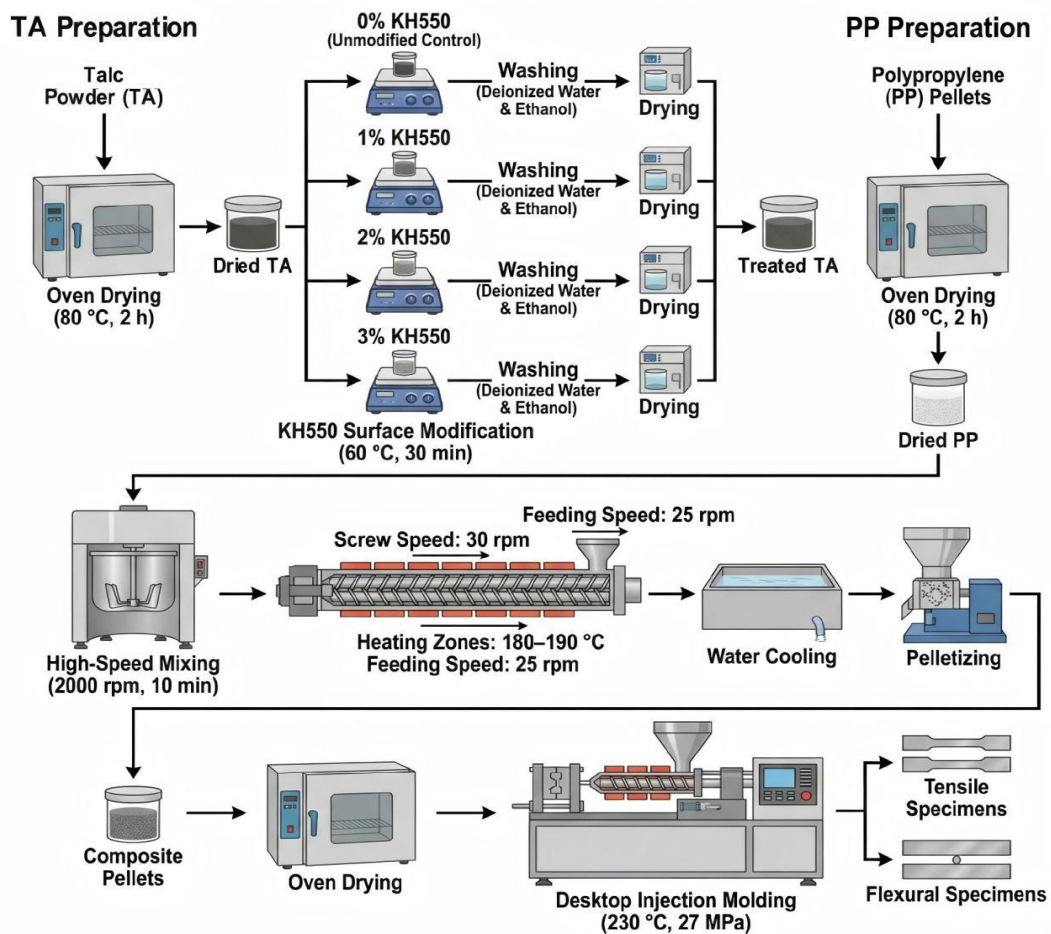


Figure 2: shows the preparation flowchart of TA surface modification and PP/UMTA and PP/MTA composites

3 Performance testing and characterization

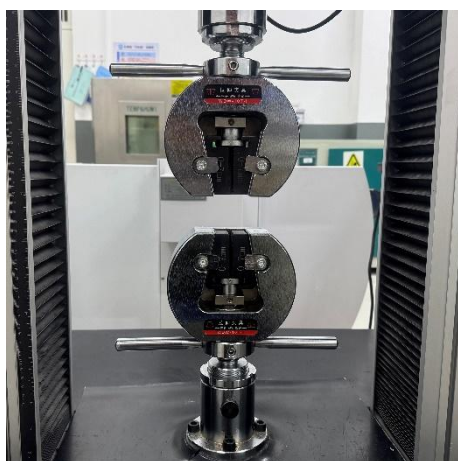
FTIR test: Fourier transform infrared spectroscopy was used to observe the changes in functional groups of talc powder. Dried potassium bromide was chosen to be mixed with the sample and pressed, the scanning range was 400-4000 cm^{-1} and the number of scans was 32.

XRD characterization: Cu target $K\alpha$ rays, scanning range 5° - 80° , scanning speed $2^\circ/\text{min}$.

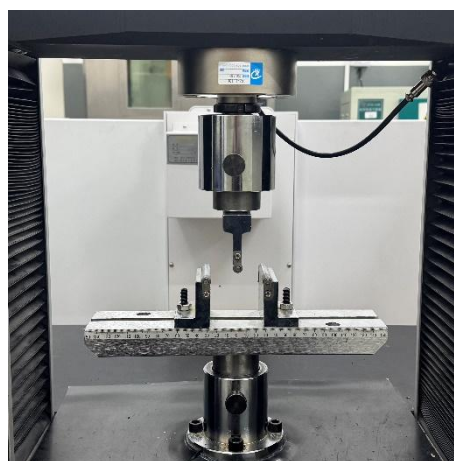
SEM analysis: Using scanning electron .0... Microscope to test the composite impact section. The prepared experimental sample strips were punched off using an impact testing machine, the impact section was protected, and the quenched section was sprayed with gold for 120s, and then observed and photographed for recording, during which the 98 accelerating voltage was 5kV.

Mechanical properties test: tensile strength and elongation at break in accordance with GB/T1040.2-2006 test, running speed 50mm/min; bending strength in accordance with GB/T9341-2008 (three-point bending) test, running speed 10mm/min.

Thermal stability test: N_2 atmosphere, set the temperature range of 30-800 $^\circ\text{C}$, the rate of temperature increase rate of 10 $^\circ\text{C} / \text{min}$.



a-Tensile test



b-Bending test

Figure 3: Experimental Equipment for Mechanical Properties Test

4 Results and Discussion

4.1 TA powder analysis

4.1.1 Water contact angle analysis

Fig. 4 shows the water contact angle comparison of for UMTA and MTA, the comparison found that the contact angle between for modified TA and water was 0° , the water droplets were flat, indicating that TA itself is hydrophilic; after KH550 modification the contact angle increased significantly from 0° to 85.423° , and TA changed from hydrophilic to hydrophobic. [6, 7] This is due to the fact that KH550 is a bifunctional coupling agent, and the chemical formula of KH550 is $-\text{NH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{Si}(\text{OC}_2\text{H}_5)_3-$, and its end group triethoxylate can hydrolyze to form silanols ($\text{Si}-\text{OH}$) in the system of water and alcohols, and then subsequently condense with the hydroxyl groups on the TA surface to form stable silicon-oxygen bonds. This process will consume and cover the hydroxyl groups on the TA surface to a certain extent and introduce organopropyl chains with organosilane-modified layers on the TA surface, thus reducing the

surface polarity, changing the surface energy, and ultimately leading to an increase in the contact angle. [8, 9]

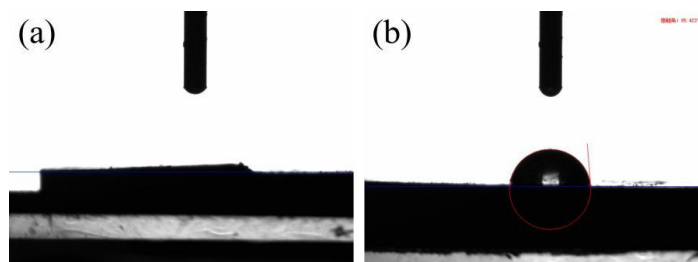


Figure 4: Contact Angle of Talc Before and After Modification

4.1.2 FTIR analysis

The infrared spectrum of modified TA is shown in Fig. 5, the molecular formula of talc (TA) is $Mg_3[Si_4O_{10}]OH_2$. In Fig. 5, the hydroxyl group (O-H) stretching vibration peaks at 3675.02 and 3423.39 cm^{-1} exist in the form of water of crystallization in TA; the silicon-oxygen bond (Si-O) stretching vibration peak at 1022.42 cm^{-1} ; and the sharp absorption band at 880.51 cm^{-1} , which is related to vertical Si-O-Mg stretching vibration. 2990-2800 cm^{-1} , and there is no significant peak near the Si-O(-1) peak. There is no obvious characteristic peak near here, theoretically this is the characteristic peak of organic matter (-CH₂), (-CH₃), so it can be judged as pure TA without organic matter. [10]

The TA modified by silane coupling agent was rinsed with ethanol for 8h, dried, and then the modified talc was pressed with potassium bromide to determine its infrared spectrum, respectively.

According to Fig. 5, the characteristic absorption peak of -NH₂- at 3428.81 cm^{-1} for MTA indicates that the amino tubulin group introduced by KH-550 was successfully grafted on the surface of the filler. Meanwhile, the characteristic peaks of -C-H-stretching vibration appeared at 2973.32 and 2888.99 cm^{-1} . This is attributed to the propyl chain -CH₂-CH₂-CH₂- structure of KH550. The modified talc surface rinsed with ethanol still retained the above organic characteristic peaks, suggesting that KH550 is not simply physically adsorbed, but is more likely to form a more stable bonding layer through condensation with surface hydroxyls, which leads to the success of TA surface modification [11].

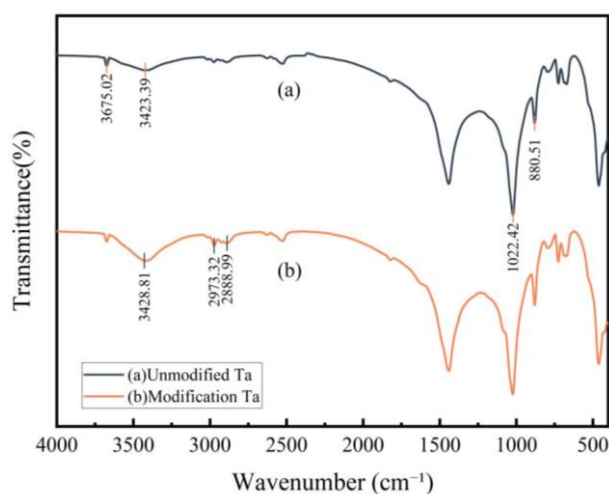


Figure 5: FTIR spectra of talc powder before and after modification

4.1.3 XRD analysis

The x-ray diffraction pattern of TA before and after modification is shown in Figure 6. As can be seen from the figure, the positions of the diffraction peaks of TA before and after modification are basically the same, and the characteristic diffraction peaks of talc crystals appeared at 2θ of 9.47° , 20.91° , 16.68° , 28.64° , 31.01° , 41.19° , 45.01° , and 51.14° , and there were no new diffraction peaks of the TA modified with different concentrations of KH550, and there was no obvious diffraction peak intensity. There was no obvious change in the intensity of the diffraction peaks. This indicates that the modified TA maintains the complete phase structure of talc and the crystal structure is not changed. [12, 13] This indicates that the organic group introduced by KH550 did not change the internal structure of TA but only coated the surface of TA. Therefore, it can be concluded from the XRD analysis that the surface modification of TA by silane coupling agent (KH550) does not affect its original internal structure. [14]

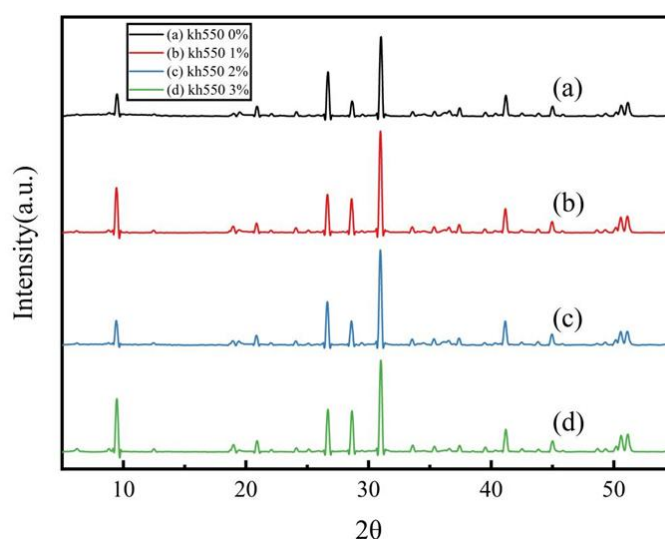


Figure 6: XRD Patterns of TA Modified with Different Concentrations of KH550 Modifier

4.2 Analysis of PP/UMTA and PP/MTA composites

4.2.1 SEM microanalysis of PP/UMTA composites

Figure 7 shows the SEM micrographs with different TA contents. (a) The TA content is 5%, and it can be seen from figure (a) that the fracture is dominated by cracks and fissures, and the surface is scattered with fine, small amount of TA particles, so the TA contribution to the composite material is relatively small, and the fracture is still dominated by the PP matrix. In (b), the TA content is 10%, the section shows more obvious flaky and laminated structure, the section is more rough and continuous, and the TA particles are dispersed more uniformly and dispersed in the matrix. (c) with a TA content of 15% shows holes in the cross-section and more flaky fragments, and these holes are the source of stress concentration. (d) with a TA content of 20% sections show larger, more irregular blocky areas, with agglomerates of larger particles and interfacial cracks around the agglomerates. [15] Comprehensive morphological characteristics, the section with 10% TA content shows the most obvious roughness without significant holes and agglomerates, which is conducive to the transfer and dissipation of stresses in the matrix.

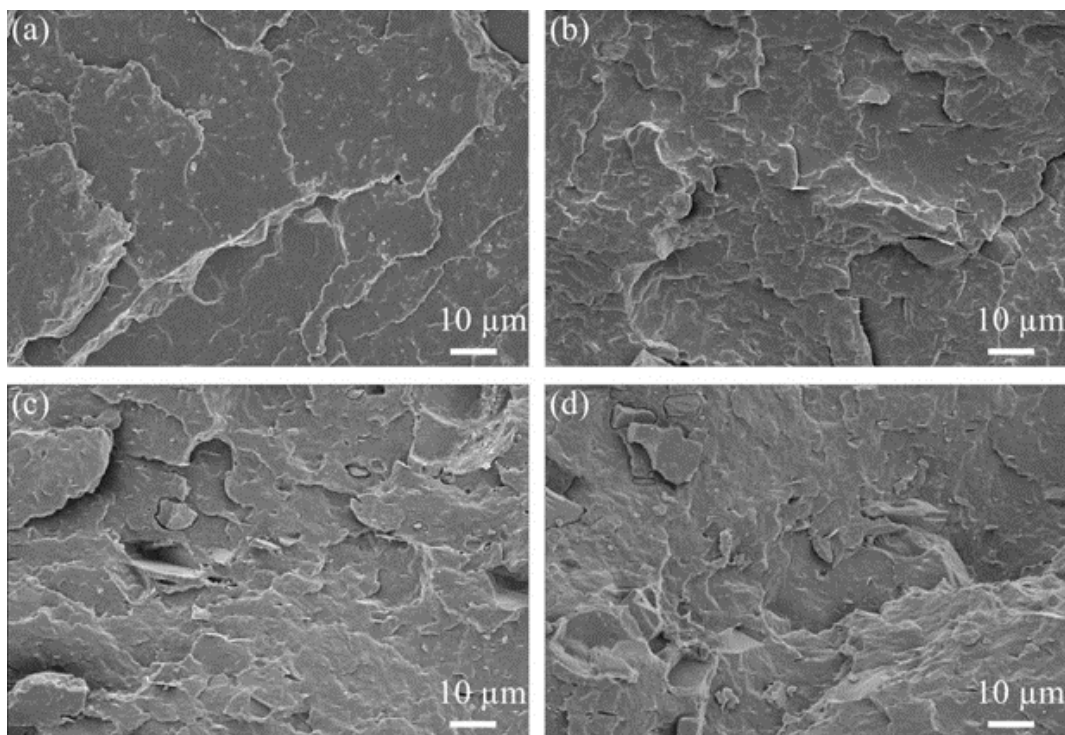


Figure 7: SEM images of PP/UMTA composites at different talc powder contents

4.2.2 SEM microscopic analysis of PP/UMTA composites

Figure 8 shows the SEM images of PP/UMTA and PP/MTA composites at different KH550 concentrations. Figures (a)(b)(c) show the composite mixtures of unmodified talc powder (TA) and polypropylene (PP), which shows that there are obvious agglomerates of PP/UMTA composites with uneven distribution of TA in the PP matrix, and obvious black crevices can be seen around the TA particles. continuous black gaps that clearly separate the TA from the PP matrix. Figures (d)(c)(f) show the composite mixture of PP/MTA with KH550 modifier concentration of 2%, it can be observed that the TA particles are uniformly dispersed in the PP matrix, and there is no gap between TA and PP matrix, the PP matrix wraps around the TA particles, and the stress whitening and plastic deformation appear in the cross section, which indicates that the stresses are successfully transferred from the matrix to the filler to form a perfect transition layer. [16]

The composite mixture of PP/MTA when the concentration of KH550 modifier is 3% is shown in Fig. (g)(h)(i), and it can be observed that TA particles are no longer uniformly distributed when the concentration of KH550 is too high, and form agglomerates with larger size and dense structure, forming irregular pores. The reason for this phenomenon is that after the active sites on the TA surface are saturated, it is difficult for the excess KH550 to further condense with the hydroxyl groups on the surface. Instead, hydrogen bonding and van der Waals forces form a multilayer physisorption accompanied by a -Si-O-Si- self-condensation network, which induces particle-bridging agglomerations; [17] This defective region is prone to be a source of cracks and lead to early failure when loaded. In conclusion, 2% KH550 is a more suitable amount of modifier for this system.

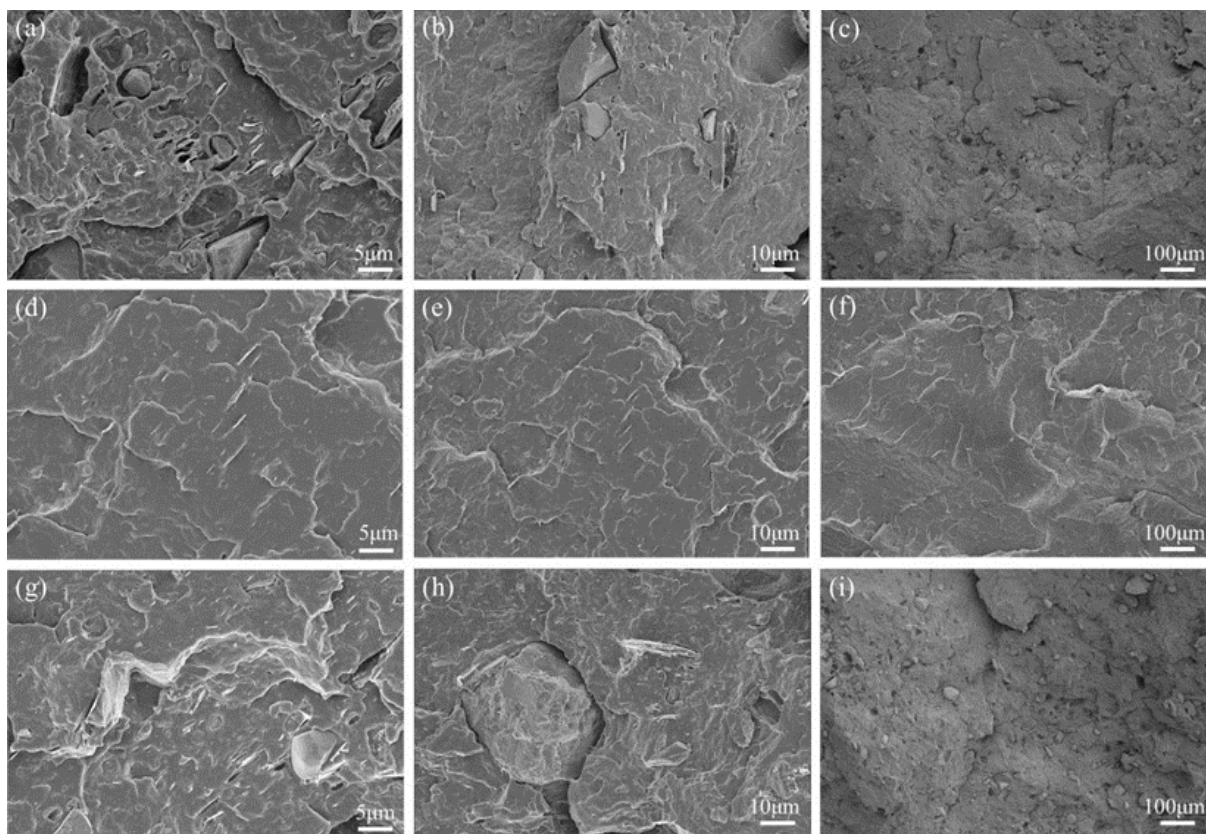


Figure 8: SEM images of modified PP/MTA composites at different KH550 concentrations

4.2.3 FTIR analysis of composites

From Figure 9(a)(b)(c), it can be seen that a telescopic vibrational peak corresponding to $-CH_2-$ is found at 2952.42 cm^{-1} for PP. Meanwhile, two bending vibrational peaks of $-CH_2-$ and $-CH_3-$ were found at 1456.42 cm^{-1} and 1375.01 cm^{-1} , and two rocking vibrational peaks of $-CH_3-$ appeared at 1162.65 cm^{-1} and 973.26 cm^{-1} . This result indicates that the prepared composites of PP/UMTA and PP/MTA did not have the main chain structure of PP unchanged by the introduction of TA and KH550.

After filling unmodified talc (UMTA) into polypropylene (PP) by Fig. 3(b), the telescopic vibration peak of $-Si-O-Si-$ was found to appear at 1026.37 , because the talc (TA) composition contained SiO_2 , and at the same time, the Si-O bending vibration peak was found at 462.37 cm^{-1} , and the appearance of these peaks, which indicated that the TA existed stably in PP, indicated that the composites of TA and PP were prepared.

Observing (c) and (b) in Fig. 9 (c) for PP/MTA and (b) for PP/UMTA comparisons revealed $-Si-O-Si-$ stretching vibrational peaks at 1026.37 cm^{-1} and Si-O bending vibrational peaks at 462.24 cm^{-1} , which can be seen from the figure that the peaks and the area of the peaks increased, which suggests that the hydrolysis of KH550 generates $-Si-OH-$ after hydrolysis of KH550 and further condensation to form $-Si-O-Si-$ network, MTA and PP are not simply physically blended. Instead, it is more likely to achieve stronger interfacial bonding and more stable structural integration through the "filler surface silanization-interfacial transition layer", which indicates that PP/MTA composites have been successfully prepared and the interfacial compatibility has been improved.

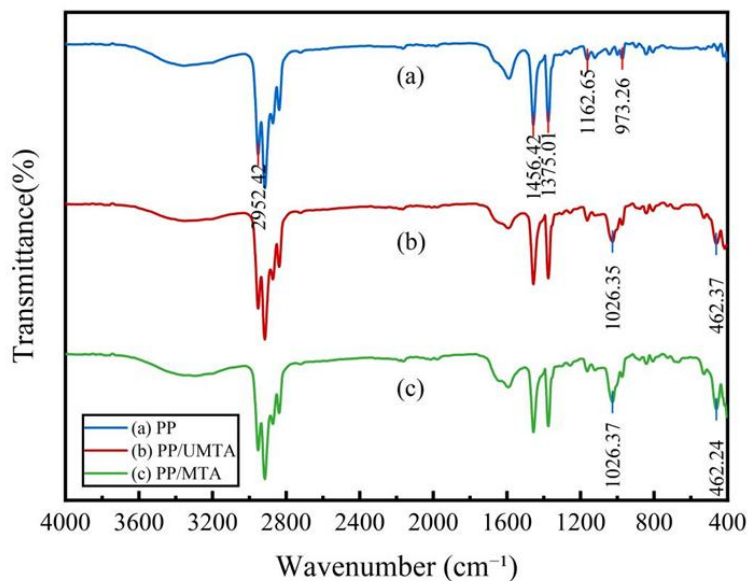


Figure 9: shows the FTIR spectra of PP, PP UMTa, and PP MTa.

4.2.4 TG-DSC analysis of composites

(1) Composite PP/UMTA analysis

Fig. 10 shows the curves of TG of composite PP/UMTA, Fig. 11 shows the curves of DTG of composite PP/UMTA, and Table 3 shows the collation of TG-DSC of composite PP/UMTA. The thermal decomposition of PP and PP/UMTA composites under the condition of N₂ atmosphere and 10°C/min heating up to 800°C is mainly manifested as a single main cracking process: the TG of the main weight loss is concentrated at ~350-500 °C, and DTG corresponds to the appearance of a single main peak ($T_{max} \approx 453-460$ °C). [18] The overall upward shift of the residential cracking peak (DTG T_{max}) to 458-460 °C compared with pure PP is 4.6-10.5 °C higher, indicating that the lamellar TA delays the appearance of the peak temperature of the maximum weight loss by altering the conditions of heat-mass transfer through increasing the cracking product, thus improving the thermal stability of the main cracking stage. The T_{max} reaches its maximum at TA (15%) It slightly decreases at TA(20%), suggesting that the barrier enhancement with filler agglomeration, interfacial defects and changes in heat transfer paths at high doping rate make the peak temperature nonlinear with respect to the doping rate. [19]

With the increase of TA content, the weight loss at 0-500°C decreased from 95.5%(5%)-90.2%(10%)-86.0%(15%)-83.0%(20%) step by step, which was mainly originated from the decrease of the mass fraction of crackable PP. Meanwhile, the weight loss in the 500-800°C tail section increases from 1.8%(5%)-2.9%(10%)-4.2%(15%)-5.0%(20%), and the DTG exhibits a gradual enhancement of the main peak, which is reflected in the limited escape of cracking products and the increased contribution of the slow secondary process. The final 800°C residue increased with doping (2.7%-6.9%-9.8%-12.0%), reflecting the increase of inorganic filler residue and high temperature stabilization residue.

The addition of UMTA in the premise of not changing the PP "single-peak main cracking" characteristics, to achieve the T_{max} overall upward shift, high-temperature residual increase, showing the TA lamellar barrier brought about by the enhancement of the basic thermal stability; however, in the high doping of the peak temperature is nonlinear change, so improve the dispersion, reduce the agglomeration of the composite thermodynamic stability is particularly important. It is especially important for the thermodynamic stability of composites.

Table 3: shows the TG-DTG data for different talc contents

	TA (wt%)	0-500°C Weight loss (%)	800°C Total weight loss (%)	800°C Residual carbon (%)	500-800°C Tail section weight loss (%)	Tmax (°C)
(a)	PP	99.7	99.8	0.2	0.1	453.5
(b)	5	95.5	97.3	2.7	1.8	458.1
(c)	10	90.2	93.1	6.9	2.9	460.5
(d)	15	86.0	90.2	9.8	4.2	464.0
(e)	20	83.0	88.0	12.0	5.0	460.2

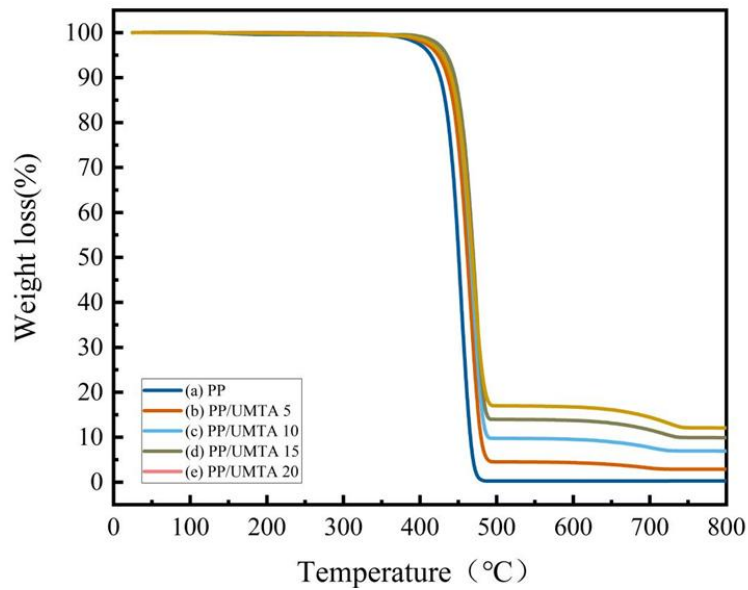


Figure 10: shows the TG curves at different talc contents

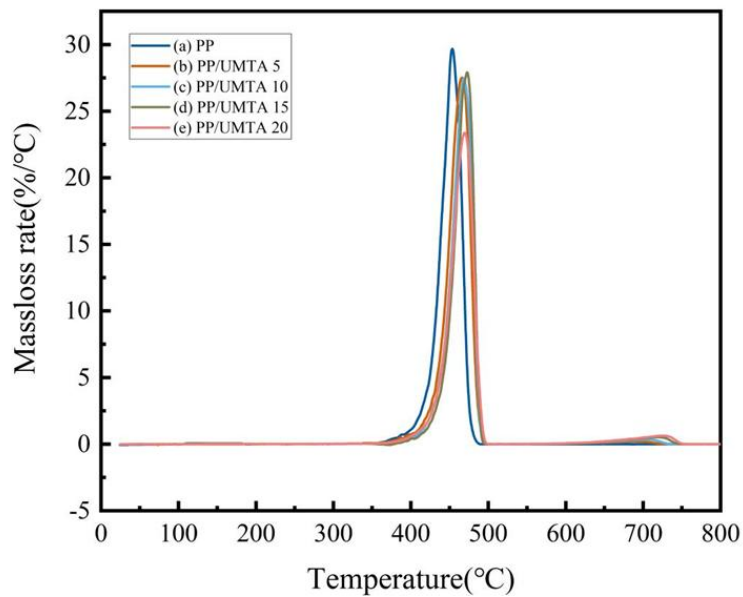


Figure 11: shows the DTG curves at different talc contents.

(2) Composite PP/MTA analysis

Composites at high doping of TA, the system because more prone to agglomeration and

interfacial porosity easily lead to the failure of the barrier network, so the introduction of KH550 in the TG-DTG analysis of the effect is significant, so the selection of TA content of 20% of the PP/MTA as the analysis of the concentration of KH550.

Fig. 12 shows the curve of TG of composite PP/MTA, Fig. 13 shows the curve of DTG of composite PP/MTA, and Table 4 shows the collation of TG-DSC of composite PP/MTA. Under the condition of N₂ atmosphere and 10°C/min heating up to 800°C, the thermal decomposition of PP and PP/UMTA composites still mainly showed a single cleavage process, and KH550 was introduced and used for the change of DTG single cracking process. introduction and for changing the basic characteristics of the DTG single peak. The T_{max} of unmodified TA was 460.2°C, while that of KH5501/2/3% was 463.6/465.2/458.1°C, in which the T_{max} of 2% was the highest for the composite system, accompanied by higher residual amount, indicating that the composite system was more prone to agglomeration and poor interfacial encapsulation under high doping, and the appropriate amount of KH550 could significantly improve the dispersion and interfacial integrity, thus amplifying the DTG peak. The moderate amount of KH550 can significantly improve the dispersion and interfacial integrity, thus amplifying the barrier network of TA lamellae with poor interfacial encapsulation; [20] However, the excessive amount of KH550 may be due to the self-condensation or the organic layer is too thick, and the secondary agglomeration and lead to the decrease of the barrier efficiency and the decrease of the T_{max}. So KH550 concentration of 2% is the optimal ratio.

Table 4: TG-DTG data at different KH550 concentrations with talc content of 20 (wt%).

	KH550 (%)	0-500°C Weight loss (%)	800°C Total weight loss (%)	800°C Residual carbon(%)	500-800°C Tail section weight loss(%)	T _{max} (°C)
(a)	PP	99.7	99.8	0.2	0.1	453.5
(b)	0	83.0	88.0	12.0	5.0	460.2
(c)	1	80.9	86.7	13.3	5.8	463.6
(d)	2	77.6	84.0	16.0	6.4	465.2
(e)	3	81.5	86.9	13.1	5.4	458.1

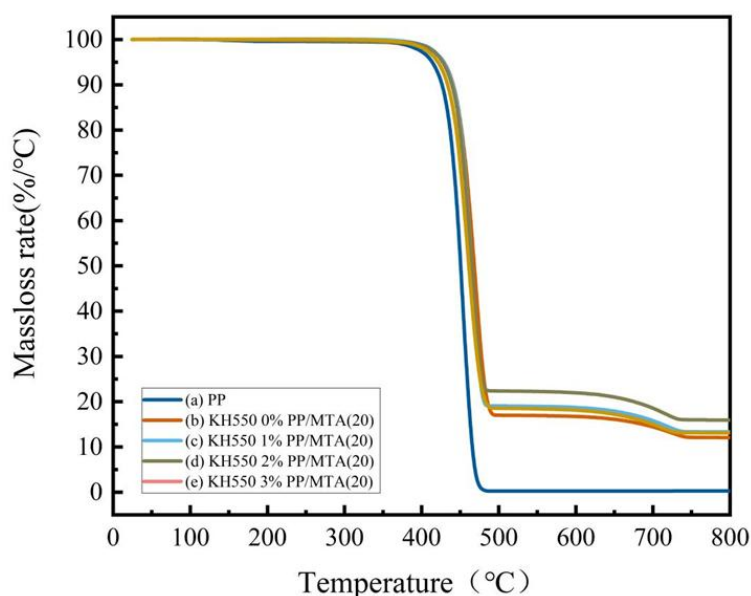


Figure 12: TG curves at different KH550 concentrations with a talc content of 20 (wt%)

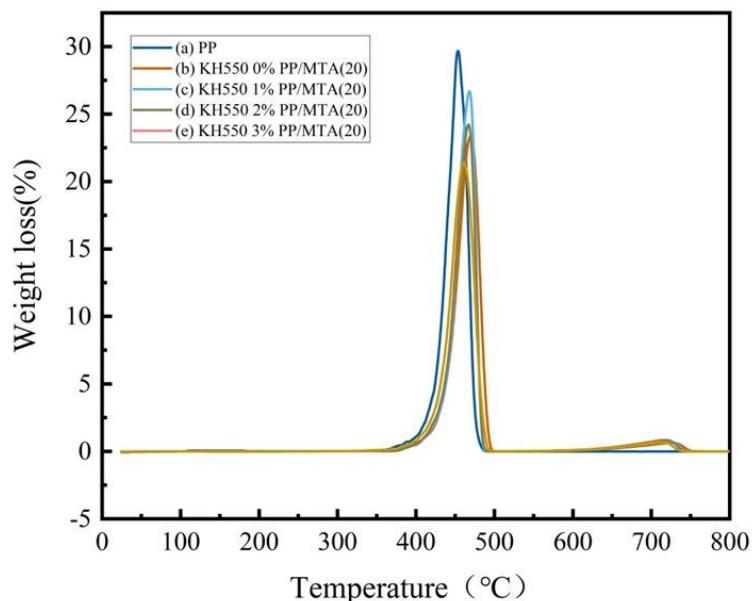


Figure 13: shows the DTG curves at different KH550 concentrations with a talc content of 20 wt%.

4.3 Mechanical properties of composites

4.3.1 Tensile test

Figure 14 shows the tensile strength of composites made with different KH550 modifier concentrations for different TA dosage. The tensile strength increases with the increase of silane coupling agent concentration when KH550 is increased from 0% to 1% and 2% at the same TA doping level. At 2% KH550 and 10% TA doping, the tensile strength of PP/MTA composites was optimized at 32.83 MPa, which is 149% higher than that of pure PP and 126% higher than that of the unmodified control. The best tensile strength performance was obtained when KH550 was 2%. This enhancement is attributed to the fact that the main component of TA is silica and contains hydroxyl groups on the surface, so when a certain amount of coupling agent is added, the ethoxyl and amino functional groups in the silane coupling agent KH550 can bind TA to the PP matrix. As the amount of silane coupling agent added increases, the coupling agent reacts chemically with the hydroxyl groups on the surface of the slip TA to form a silicone-oxygen layer on the surface of the TA, which will improve the compatibility and wettability of the TA. can improve the dispersion of TA in PP and improve the interfacial adhesion between the two, which leads to the enhancement of the interaction force between TA and PP, so its mechanical properties are improved. The macroscopic manifestation is the tensile strength enhancement.

When KH550 is further increased to 3%, the tensile strength falls back ("first increase then decrease"). This phenomenon usually corresponds to an interfacial failure mechanism caused by an excess of coupling agent: this is due to the fact that the reaction centers in the TA filler are fully occupied and the reaction sites are saturated, and instead of reacting with the filler, the new KH550 is more likely to self-condense to form an oligomeric mesh of -Si-O-Si- and to build up in a physisorptive manner to form an organosilane rich layer or a filler-filler "bridging aggregation layer". [21] This layer tends to have low cohesive strength and low modulus, and is prone to interfacial layer cohesion damage or induced microporous defects under tensile loading, which weakens the interaction between TA and PP, thus weakening the effective stress transfer and leading to a reduction in strength. [22]

When KH550 is fixed, the tensile strength increases from 5% to 10% and reaches a better level. TA is a rigid filler with a high aspect ratio, which can form an effective load-bearing phase at low to medium dosage: this is due to its rigidity and orientation effect that improves the material's ability to withstand tensile loads, and on the other hand, the right amount of filler can promote a more even dispersion of the stresses in the matrix, which leads to a strength gain. Strength gain.

When the TA is further increased to 20% (high doping), the tensile strength shows a drop back. The essence is that the competition between the "enhancement effect" and the "defect effect" is tilted towards the latter at high dosage: a high filler volume fraction significantly increases the filler-filler contact probability, which leads to secondary agglomeration and an increase in interfacial voids; at the same time, the continuous phase of the matrix is weakened, and the melt coating is difficult. weakened, melt encapsulation difficulty, local stress concentration enhancement, and it is easier to start cracking from agglomerate-interface defects and expand rapidly during tensile, which is finally manifested in the decrease of tensile strength with the increase of TA.

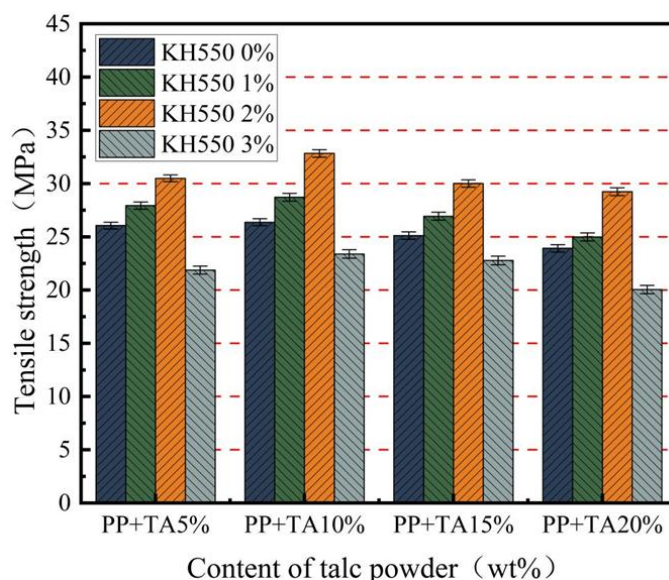


Figure 14: Tensile strength of PP/TA composites modified with different KH550 modifier concentrations

4.3.2 Bending test

Figure 15 shows the flexural strength of PP/TA composites modified with different concentrations of KH550 for different TA dosage. The flexural strength increases with the increase of silane coupling agent concentration when KH550 is increased from 0% to 1% and 2% at the same TA dosage. The optimum flexural strength of PP/MTA composites with 20% TA doping at 2% KH550 is 48.32 MPa compared to pure PP with 127% increase and 112% increase in tensile strength compared to unmodified control. As shown in Fig. X, the flexural strength of PP/TA composites was determined by both talc loading and KH550 dosage. Increasing the TA content usually enhances the flexural strength, which is attributed to the rigid platelet talc machine that effectively withstands flexural stresses and enhances the load-bearing capacity of the composites. KH550 exhibits the best dosage: its flexural strength is significantly enhanced from 0 to 2%, indicating wettability, improved dispersion properties, and enhanced interfacial stress transfer, as well as a reduction in interfacial debonding defects that usually trigger tensile-side cracks in bending. However, further enhancement of KH550 to 3% leads to

diminishing or even slightly decreasing gains due to the fact that excess silanes may form a polarity-rich network between particles, which induces secondary agglomerations, and the agglomerates become a source of cracks on the tensile side of bending. Also the excess amino (-NH₂) and alkyl long chain (-CH₂-CH₂) -interfacial layer is more hygroscopic and the stressing process is more likely to induce microporous defects.

At KH550 fixation, the overall bending strength shows an upward trend with the increase of TA from 5% to 20%, this is due to the fact that the damage in bending test is often dominated by microcracks sprouting on the tensile side, and also the overall load bearing is related to the stiffness modulus of the material. The TA (lamellar stiffening filler) raises the modulus of the system, so that a higher load is required for the same deflection, which is often manifested as a rise in the bending strength. TA flakes are easily oriented under processing shear (parallel to the flow direction), and under bending loading this orientation is more likely to form a "laminar-like load", which usually contributes more to bending strength than to tensile strength. So the bending strength increases with increasing TA.

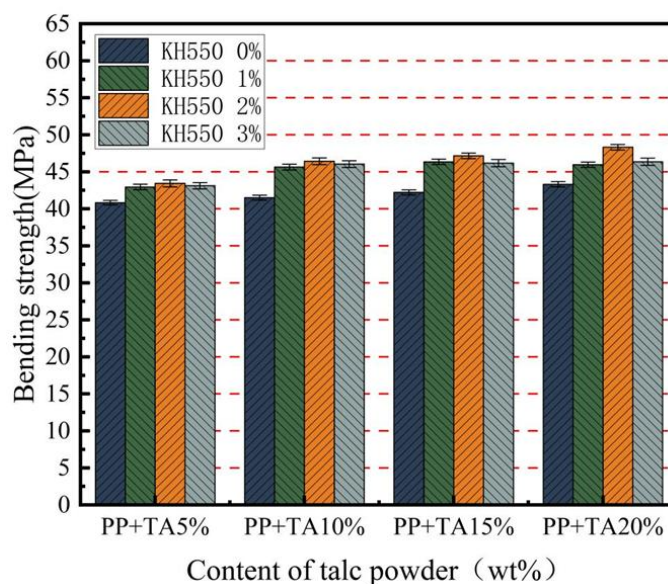


Figure 15: Flexural strength of PP/TA composites modified with different KH550 modifier concentrations

5 Conclusion

(1) The surface energy difference and thermodynamic incompatibility between unmodified TA and PP lead to poor dispersion and interfacial debonding, which is prone to stress concentration and limits the reinforcement efficiency.

(2) The hydrolytic condensation of KH550 can form a stable coating layer on the surface of TA, increase the degree of filler surface organicization and PP melt wettability, improve the dispersion and enhance the interfacial bonding, so as to enhance the tensile and flexural properties of the composites; among which, 2% of KH550 exhibits a better synergy between interfacial and dispersion effects.

(3) The mechanical properties were as follows: low-moderate TA content was favorable to the formation of effective load-bearing phase and strength enhancement, while high filler content was easy to induce secondary agglomeration and pore defects, leading to a drop in tensile properties; the bending properties were more sensitive to modulus enhancement and orientation load-bearing, and the overall enhancement was more significant with the increase

of TA content.

(4) Excessive amount of KH550 (3%) can easily lead to particle-bridging agglomeration and introduction of defective holes due to multilayer adsorption-Si-O-Si-self-condensation, which can weaken the interfacial stress transfer and cause performance degradation.

Funding

This work was supported by Department of Science and Technology of the Ningxia Hui Autonomous Region and the Research Fund of North Minzu University (2020KYQD40).

References

- [1] Goswami R, Khosla A, Alam A, et al. Experimental investigation on the spectral, mechanical, and thermal behaviors of thermoplastic starch and delaminated talc-filled sustainable bio-nanocomposite of polypropylene. *Journal of the Mechanical Behavior of Materials*, 2025, 34.
- [2] QIU F, WANG M, HAO Y, et al. The effect of talc orientation and transcrystallization on mechanical properties and thermal stability of the polypropylene/talc composites. *Composites Part A: Applied Science and Manufacturing*, 2014, 58: 7-15.
- [3] Castillo A L, Barbosa E S, Capiati J N. Influence of talc genesis and particle surface on the crystallization kinetics of polypropylene/talc composites. *Journal of Applied Polymer Science*, 2012, 126 (5): 1763-1772.
- [4] Wu, Jyh-Horng, Chen, et al. Mechanical properties, morphology, and crystallization behavior of polypropylene/elastomer/talc composites. *Polymer Composites*, 2015, 36.
- [5] ASHENAI GHASEMI F, GHASEMI I, MENBARI S, et al. Optimization of mechanical properties of polypropylene/talc/graphene composites using response surface methodology. *Polymer Testing*, 2016, 53: 283-292.
- [6] WANG L, SCHUBERT U S, HOEPPENER S. Surface chemical reactions on self-assembled silane based monolayers. *Chemical Society Reviews*, 2021, 50(11): 6507-6540.
- [7] QUÉRÉ D. Wetting and Roughness. *Annual Review of Materials Research*, 2008,(1): 71-99.
- [8] JING M, ZHANG FAN Z, et al. Markedly improved hydrophobicity of cellulose film via a simple one-step aminosilane-assisted ball milling. *Carbohydrate Polymers*, 2021, 275: 118701.
- [9] SHANG X, ZHU Y, LI Z. Surface modification of silicon carbide with silane coupling agent and hexadecyl iodide. *Applied Surface Science*, 2017, 394: 169-177.
- [10] ZHANG M, REDFERN S A T, SALJE E K H, et al. Dehydroxylation, proton migration, and structural changes in heated talc: An infrared spectroscopic study. *American Mineralogist*, 2006, 91(5-6): 816-825.
- [11] WEI B G, CHANG Q, BAO C X, et al. Surface modification of filter medium particles

- with silane coupling agent KH550. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 2013, 434: 276-280.
- [12] ZHAO J, GAO T, DANG J, et al. Using green, economical, efficient two-dimensional (2D) talc nanosheets as lubricant additives under harsh conditions. *Nanomaterials*, 2022, 12(10): 1666.
- [13] FIORENTINO B, FULCHIRON R, BOUNOR-LEGARÉ V, et al. Chemical modification routes of synthetic talc: Influence on its nucleating power and on its dispersion state. *Applied Clay Science*, 2015, 109-110: 107-118.
- [14] LIU M, LIU B, ZHAO P, et al. Silane coupling agent enable two-dimensional layered binary composite to synergistic enhance the tribological performance in grease additive. *Chemical Engineering Journal*, 2024: 158640.
- [15] ŚWIETLICKI M, et al. The structure and mechanical properties of the surface layer of polypropylene polymers with talc additions. *Materials*, 2020, 13(3): 698.
- [16] CHO J, et al. Review: 3-Aminopropyltriethoxysilane (APTES) deposition methods on oxide surfaces in solution and vapor phases for biosensing applications. *Biosensors*, 2023, 13(1): 36.
- [17] TSIOTPSIAS C, LEONTIADIS K, NTAMPOU X, et al. Modification of talc and mechanical properties of polypropylene-modified talc composite drawn fibers. *Journal of Composites Science*, 2024, 8(3): 91.
- [18] ESMIZADEH E, TZOGANAKIS C, MEKONNEN T H. Degradation behavior of polypropylene during reprocessing and its biocomposites: thermal and oxidative degradation kinetics. *Polymers*, 2020, 12(8): 1627.
- [19] WANG Y, LIU X, GIJSMAN P. Effect of talc content on the degradation of re-extruded polypropylene/talc composites. *Polymer Degradation and Stability*, 2013, 98(11): 2278-2285.
- [20] HU Y, LIU Y, ZHENG S, KANG W. Progress in application of silane coupling agent for clay modification to flame retardant polymer. *Molecules*, 2024, 29(17): 4143.
- [21] YE Y, ZHANG W, ZHANG X, et al. Preparation and characterization of reed biochar/polypropylene composites: Effects of coupling agent content and effects of coupling agent content and particle size on properties. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 2024, 703: 135325.
- [22] CAPIATI N J, PORTILLO R A, STAMBOULIS A, et Influence of talc morphology on the mechanical properties of talc-filled polypropylene injected composites. *Journal of Polymer Research*, 2013, 20: 152.