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A novel technology for powder dispersion and surface modification

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Abstract A novel technology for powder dispersion and surface modification was studied by experiment in this paper. In the process, powders are dispersed by the nozzle and the atomized modifying agent is sprayed into the dispersed particles and coated on the surface at the same time. Silica, mica and talc powders were dispersed and modified by this technology, the performance of powder dispersion is evaluated by the scanning electron microscope (SEM), and the performance of surface modification is evaluated in advance by the viscosity of powder-paraffin suspension. In experiment, the modified silica was filled in the polypropylene (PP), the impact fractographs, notched impact and tensile properties of the PP composites were studied, the results indicate that the developed technology is effective for powders dispersion and surface modification.

Introduction

Nowadays, dispersion of powder material in the air is always a critical problem, which is important in the powder technology and related industrial fields [1], such as aerosol production, pneumatic conveying and size classification. In various application fields of the powder material, different surface properties of the powder are required. So, powder dispersion and surface modification are more and more important in industrial fields recently.

People usually use mechanical, drying or electrostatic methods to achieve powder dispersion [1–5], and adopt wet, dry or vapor phase techniques to achieve the proper surface properties of the powder [6–9]. For powder dispersion, when the mechanical forces or other fluid forces used to disperse the powder, the surface properties of the powder will not be changed, the attractive forces between the particles also exist after dispersion and the particles may aggregate again. Thus, in order to avoid the powder re-aggregation, the basic and effective way for powder dispersion is to decrease or eliminate the attractive forces and decrease the surface energy of the primary particles. Surface modification is the common method to decrease the attractive forces and the surface energy of inorganic powder, which is often used for powder dispersion. If the particles are aggregated together, the modifying agent will not be coated on the surface of the core particles in aggregate particles, in order to obtain the better performance of surface modification, the powder should be dispersed firstly and the surface of every primary particle can be coated by the modifying agent. So, powder dispersion and surface modification are supplement for each other. According to the idea mentioned above, a novel technology for powder dispersion and surface modification is introduced and studied by experiment in this paper.

Principle of the technology

The principle of the technology for powder dispersion and surface modification is shown in Fig. 1. The

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Table 1 Properties of samples

Material	Density (g/cm^3)	D$_{10}$ (μm)	D$_{50}$ (μm)	D$_{90}$ (μm)	Specific surface area (m^2/g)
Quartz	2.65	2.38	3.97	6.75	1.62
Mica	2.85	2.68	5.56	11.32	1.26
Talc	2.69	4.25	12.22	29.53	0.68

aggregate particles are firstly fed into a stainless-steel pipe by a smooth screw feeder, carried by high-speed and high-temperature airflow. Thus, the particles will be dried by high-temperature air and dispersed by high-speed airflow through a nozzle. Then, the modifying agent is sprayed into the dispersed particles by an atomizing nozzle, combined with the dispersed particles to form a mixture, more interfaces between the modifying agent and the surface of particles will come into being. The temperature of the mixture and the content of modifying agent were controlled. Maintained for a few minutes, the modifying agent will be coated on the surface of the particles. At last, the modified particles are sprayed out, cooled by the atmosphere air and collected by collector.

Experiment

Experiment material

Samples and chemicals used in the present investigation include: fly-ash spheres taken from Huainan Pingwei power plant, thin-shell hollow glass spheres taken from Shenzhen microspace special materials Co., Ltd., quartz (SiO$_2$), mica and talc purchased from Beijing Dahongmen chemical material company, their main properties are listed in Table 1. Polypropylene(PP) and paraffin oil purchased from Sinopec Beijing Yanshan chemical corporation, vinyltriethoxysilane (A-151) purchased from Tianjin chemical reagent Co. plc., modifying agent γ-Aminopropyltriethoxysilane(KH-550), tetraisopropyldi(dioctylphosphate)titanate (NDZ-401) purchased from Najing Shuguang chemical group Co., Ltd.

Testing methods

Powder is dispersed and modified by the technology in Fig. 1. The viscosity of the powder-paraffin suspension is measured by rotation viscometer NDJ-1 (Shanghai Precision & Scientific Instrument Co., Ltd.), the mechanical properties of PP composites are tested by universal testing machine DXLL-1000 (No.4 Chemical Machinery Plant of Shanghai Chemical Equipment Co., Ltd.) and pendulum impact tester XJ-40A (Wuzhong Material Testing Co., Ltd.), the impact fractographs and powders are observed by the scanning electron microscope (SEM) LEO-1450 (LEO, Ltd., Germany).

The dispersion and modification processes are studied respectively in this paper. Thin-shell hollow glass spheres were used to evaluate the dispersion forces in dispersion process and silica, mica and talc powders were dispersed by the developed technology. SEM photographs of the powder before and after dispersion were used to evaluate the dispersion performance. Because of the viscosity of powder-paraffin suspension is allied to the compatibility between the powder and paraffin: lower viscosity means better compatibility at the same temperature and concentration, the viscosity of the powder-paraffin suspension could evaluate the performance of modification in advance. Finally, the modified powders were filled in PP as filler and the tensile and notched impact properties of the PP composites were studied to evaluate the performance of modification in practice.

Experiment results and discussion

Dispersion process

In order to obtain better dispersion performance, proper nozzle should be adopted in powder dispersion process. Four nozzles were chosen, the dispersion uniformity and the dispersion forces were evaluated by experiment.

The photographs of jet flow of fly-ash spheres dispersed by various nozzles are presented in Fig. 2,

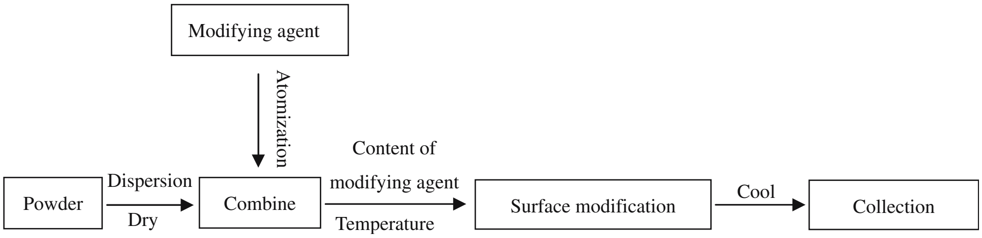


Fig. 1 Schematic graph of powder dispersion and surface modification process

which taken by digital camera SONY-F717. It could be seen that there is a bright white line in the axis of the jet flow in Fig. 2a–c, which is the concentration of numerous fly-ash spheres. At a distance, the white line becomes illegible which indicates that the concentrated fly-ash spheres are dispersed gradually. In Fig. 2d, no bright white line could be seen in the axis of the jet flow, indicating no particles concentrate on the axis of jet flow. The above results may indicate that the dispersion uniformity of particles dispersed by the internal-swirling nozzle is better than dispersed by the other nozzles.

The dispersion performance of the nozzle for powder dispersion not only includes the dispersion uniformity of aggregate particles, but also includes the dispersion forces induced by the nozzle. That is to say, the dispersion forces should be large enough to disrupt the aggregate particles. Aimed at this subject, the dispersion forces induced by various nozzles are investigated experimentally. Because it is difficult to measure the magnitude of dispersion forces directly, a new indirect method was adopted to estimate the magnitude of dispersion forces: thin-shell hollow glass spheres, which possess a density of about 0.6 g/cm^3, were chosen and dispersed by the nozzle. The magnitude of dispersion forces could be estimated by the quantity of fragmentized hollow glass spheres: more fragmentized hollow glass spheres, means the larger dispersion forces induced by the nozzle. All experiments were carried out with the following conditions: pressure of the air was 0.55 MPa, particles used in the experiment were thin-shell hollow glass spheres and the flux was 75 g/min.

The micrographs of thin-shell hollow glass spheres dispersed by various nozzles are shown in Fig. 3, which taken by optical microscope. Figure 3a shows the micrograph of original hollow glass spheres and no fragments could be seen. The micrographs of hollow glass spheres dispersed by the Laval and convergent nozzle are presented in Fig. 3b and c, in which, it could be seen that parts of the hollow glass spheres are cracked after dispersion. Figure 3d is the micrograph of the hollow glass spheres dispersed by the designed internal-swirling nozzle, from which it could be seen that all of the hollow glass spheres are cracked. Figure 3, as a whole shows that the dispersion forces induced by the designed internal-swirling nozzle are larger than induced by the Laval and the convergent nozzle.

In order to evaluate the dispersion force quantitatively, the samples of 5 g thin-shell hollow glass spheres were weighed and then fed into a 25-ml measuring cylinder, which was filled with 15 cm^3 distilled water. The samples and the water were then mixed adequately in the measuring cylinder and deposited for 20 min. Because of the density of the thin-shell hollow glass spheres is about 0.6 g/cm^3, they will float on the water, but the cracked glass spheres is not hollow, the density of the fragments is larger than the density of water and the fragments of the hollow glass spheres will deposit, less floating hollow glass spheres means more cracked hollow glass spheres, indicates the dispersion forces is larger. The quantity of hollow glass spheres floating on the water was measured and the ratio of fragments was calculated from:

$$\eta = \frac{V_1 - V_2}{V_1} \qquad (1)$$

where η is the fragments ratio, V_1 the volume of the floating hollow glass spheres in 5 g samples which were not dispersed by the nozzle, V_2 the volume of the floating hollow glass spheres in 5 g samples which were dispersed by the nozzle. The volume changes of the floating hollow glass spheres dispersed by various nozzles are shown in Fig. 4, the results of the fragments ratio are listed in Table 2, which are in reasonable agreement with the results of microscope photographs.

The above experimental results indicate that the dispersion forces induced by the designed internal-swirling nozzle are larger than other nozzles, the dispersion uniformity of particles dispersed by internal-swirling nozzle is also better than other nozzles. So, the designed internal-swirling nozzle was adopted in the dispersion process.

Silica, mica and talc powders were dispersed by the developed technology and the flowability of the powder has been enhanced after dispersion. The SEM photographs of the powder before and after dispersion are shown in Fig. 5. From the photographs it can be seen that some bigger aggregates are presented in the original silica, mica and talc powder, the morphology is rough in photo. After dispersion, there are no aggregates in the dispersed powders and the morphology is smooth in photo, which indicates that the novel technology is effective for powder dispersion.

Modification process

In order to study the influence of modifying temperature, content and kinds of the modifying agent on powder modification performance, silica was dispersed and modified by the developed technology. The viscosity of the silica-paraffin suspension was measured and plotted against the modifying temperature, content and kinds of modifying agent. In Fig. 6a, 1 wt% of KH-

Fig. 2 Photographs of jet flow of fly-ash spheres dispersed by various nozzles. (**a**) Invariable area nozzle, (**b**) Convergent nozzle, (**c**) Laval nozzle, (**d**) Internal-swirling nozzle

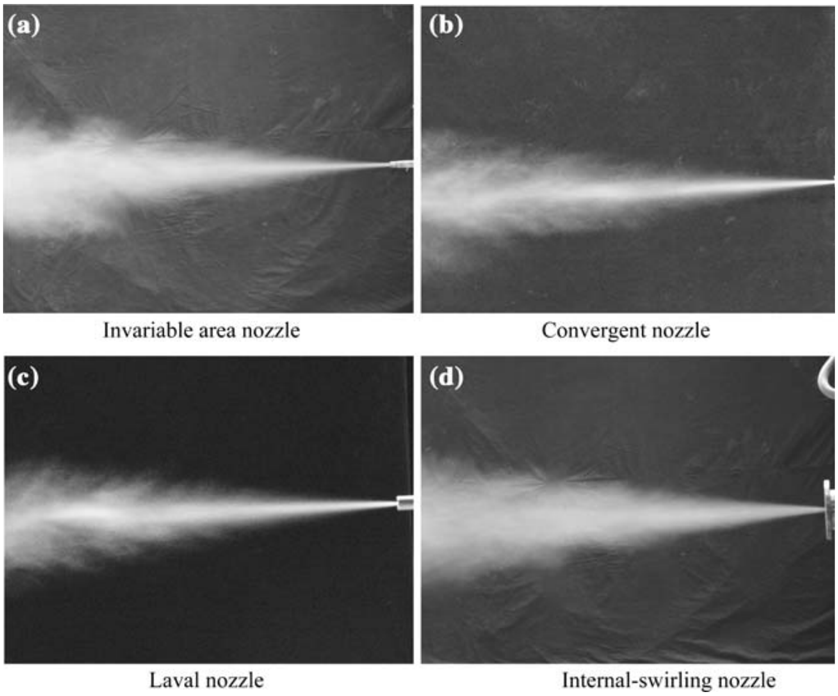


Fig. 3 The micrographs of thin-shell hollow glass spheres, dispersed by various nozzles. (**a**) original hollow glass spheres (40×), (**b**) Laval nozzle (40×), (**c**) Convergent nozzle (40×), (**d**) Internal-swirling nozzle (100×)

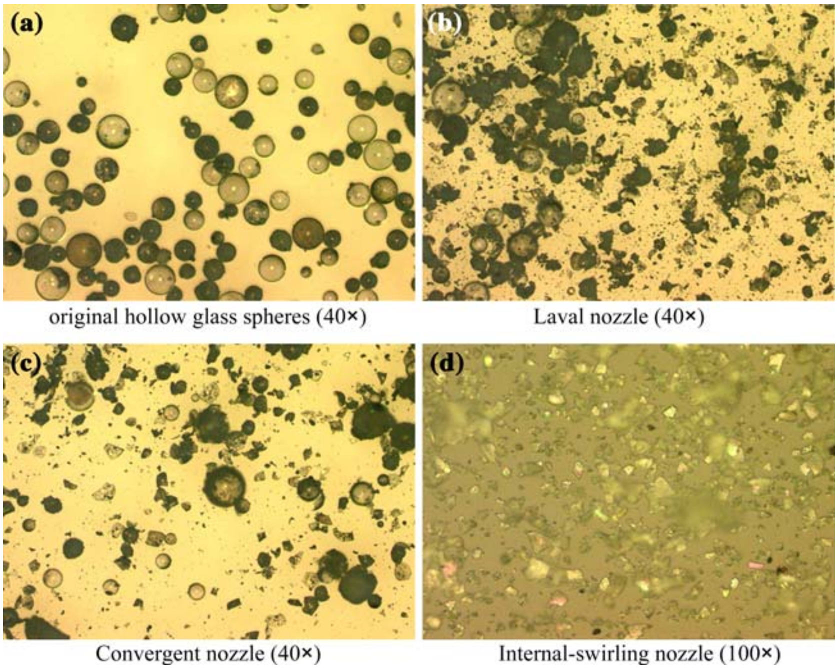


Fig. 4 The volume changes of the floating hollow glass spheres dispersed by various nozzles. (**a**) Laval nozzle, (**b**) Convergent nozzle, (**c**) Internal-swirling nozzle

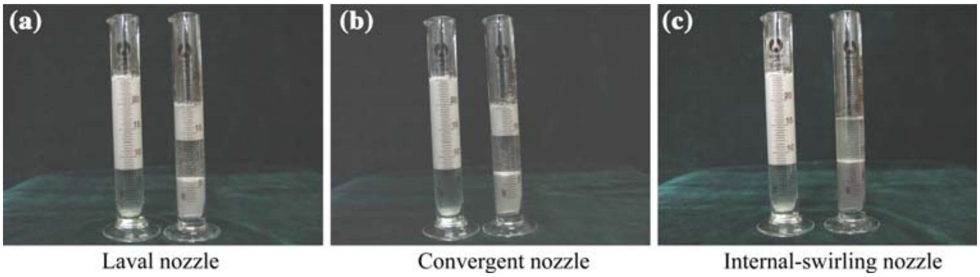
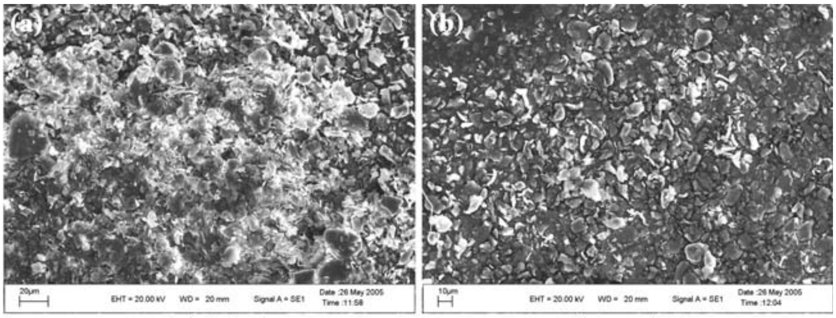


Table 2 The fragments ratio of thin-shell hollow glass spheres dispersed by various nozzles

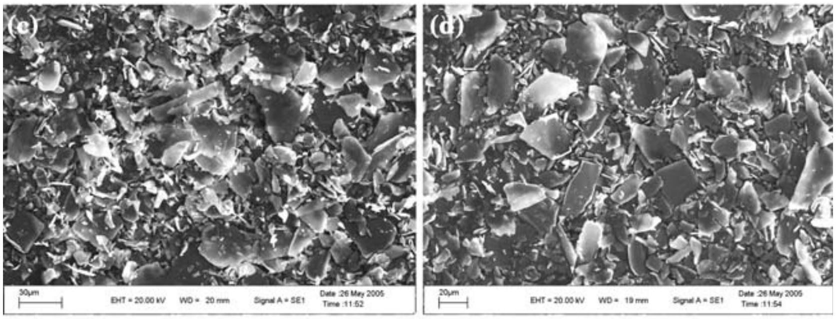
Nozzle	Fragments ratio η
Laval nozzle	61%
Convergent nozzle	68%
Internal-swirling nozzle	100%

550 is used, it can be seen that the viscosity of suspension is almost 300 mPa s at different temperature, which indicates that the modifying temperature has little effect on modification performance. In Fig. 6b, KH-550 is used at 110 °C, it can be seen that the viscosity of unmodified silica-paraffin suspension is much high as 2500 mPa s, the viscosity decreases with the content increase, and decreases to 300 mPa s when the silica is modified by 1.0 wt% of KH-550, then increases at higher content. In Fig. 6c, 1 wt% of modifying agents are used at 110 °C, the viscosity of the silica-paraffin suspension decreases more after the silica surface modification, and a minimum viscosity appears as the silica was modified by NDZ-401, which shows that the silica modified by NDZ-401 have the best compatibility with paraffin.

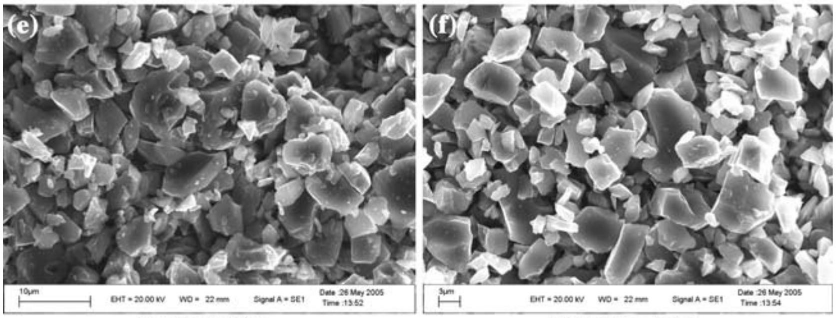
In order to convict that the molecular layer was formed, Fourier transform infrared spectroscopy (FTIR) was used to study the changes of the silica before and after modification. The FTIR data were collected using Nicolet Magna-IR 750 spectrometer. The FTIR spectra of unmodified silica, KH-550 modified silica and pure KH-550 coupling agents are presented in Fig. 7. The broad scan FTIR spectra is shown in Fig. 7a and the narrow scan FTIR spectra is shown in Fig. 7b. In the spectra of pure KH-550, there appear the characteristic absorption peaks of methylene at 2861, 2927 cm^{-1} and the stretching vibrations peaks of N–H at 3355, 3290, 3184 cm^{-1}, which exist in the spectra of KH-550 modified silica, not exist in the spectra of unmodified silica. It indicates that the functional groups of KH-550 have reacted with



Original talc Dispersed talc



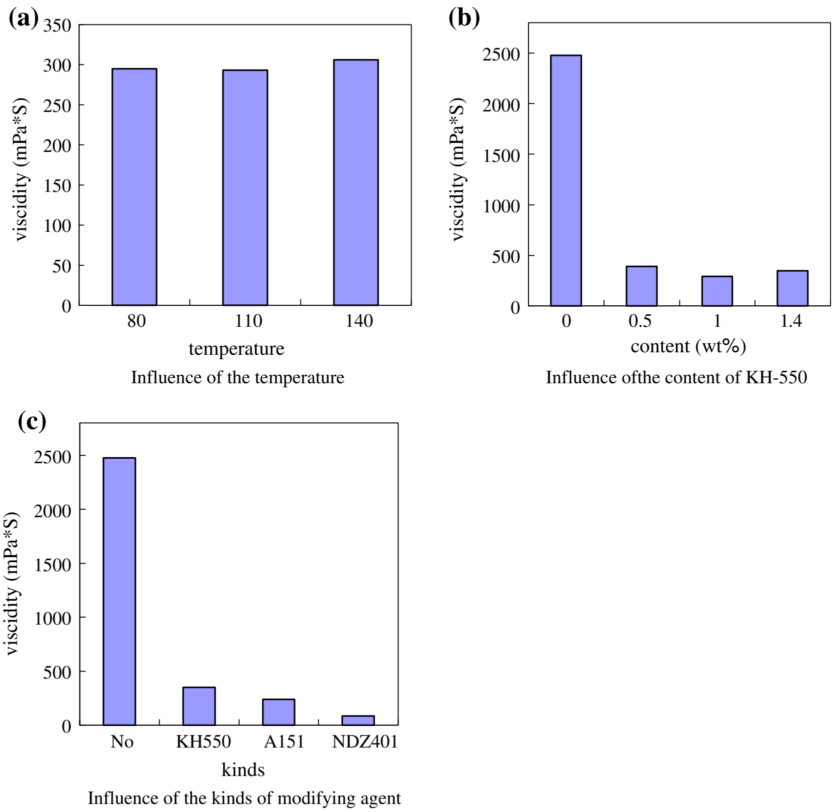
Original mica Dispersed mica



Original silica Dispersed silica

Fig. 5 SEM photographs of silica, mica and talc powders before and after dispersion. (**a**) Original talc, (**b**) Dispersed talc, (**c**) Original mica (**d**) Dispersed mica, (**e**) Original silica (**f**) Dispersed silica

Fig. 6 Influence of temperature, content and kinds of modifying agent on the viscosity of silica-paraffin suspension, the silica content is 50 wt%. (**a**) Influence of the temperature, (**b**) Influence of the content of KH-550, (**c**) Influence of the kinds of modifying agent



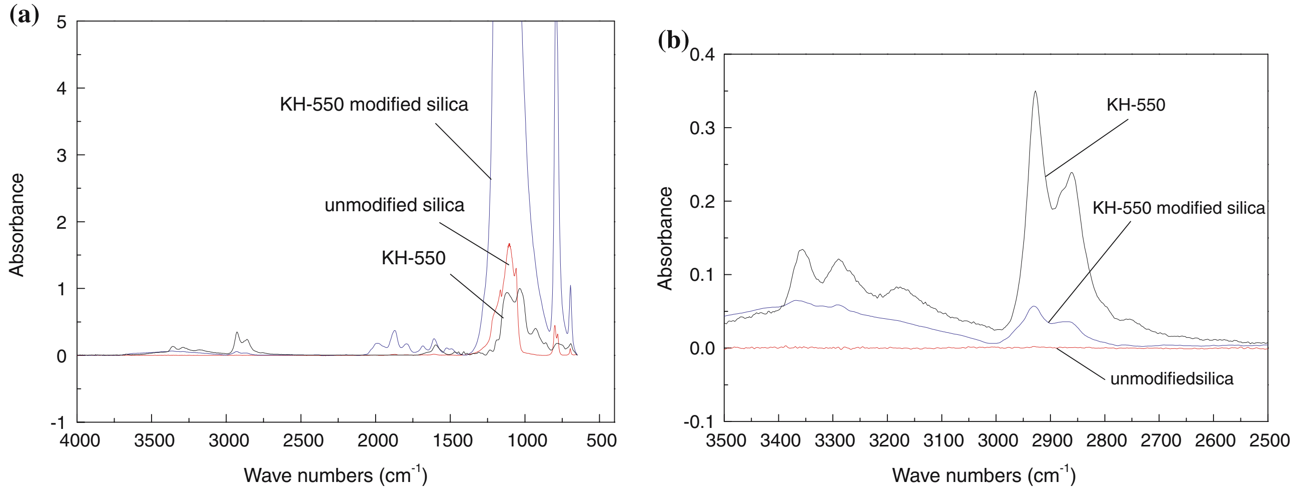


Fig. 7 FTIR absorption spectra of unmodified silica, KH-550 modified silica and pure KH-550, (**a**) The broad scan FTIR spectra of unmodified silica, KH-550 modified silica and pure KH-550, (**b**) The narrow scan FTIR spectra of unmodified silica, KH-550 modified silica and pure KH-550

hydroxyl group on the surface of silica and the coupling agent molecules are bonded to the surface to form an organic layer.

The viscosity of silica-paraffin suspension is usually utilized to evaluate the modification performance in advance. In industry, the practical performance is utilized to evaluate the modification performance. So, the modified silica was filled in PP as filler in our experiment, the tensile and notched impact properties of the PP composites were studied to evaluate the modification performance and the results are shown in Fig. 8.

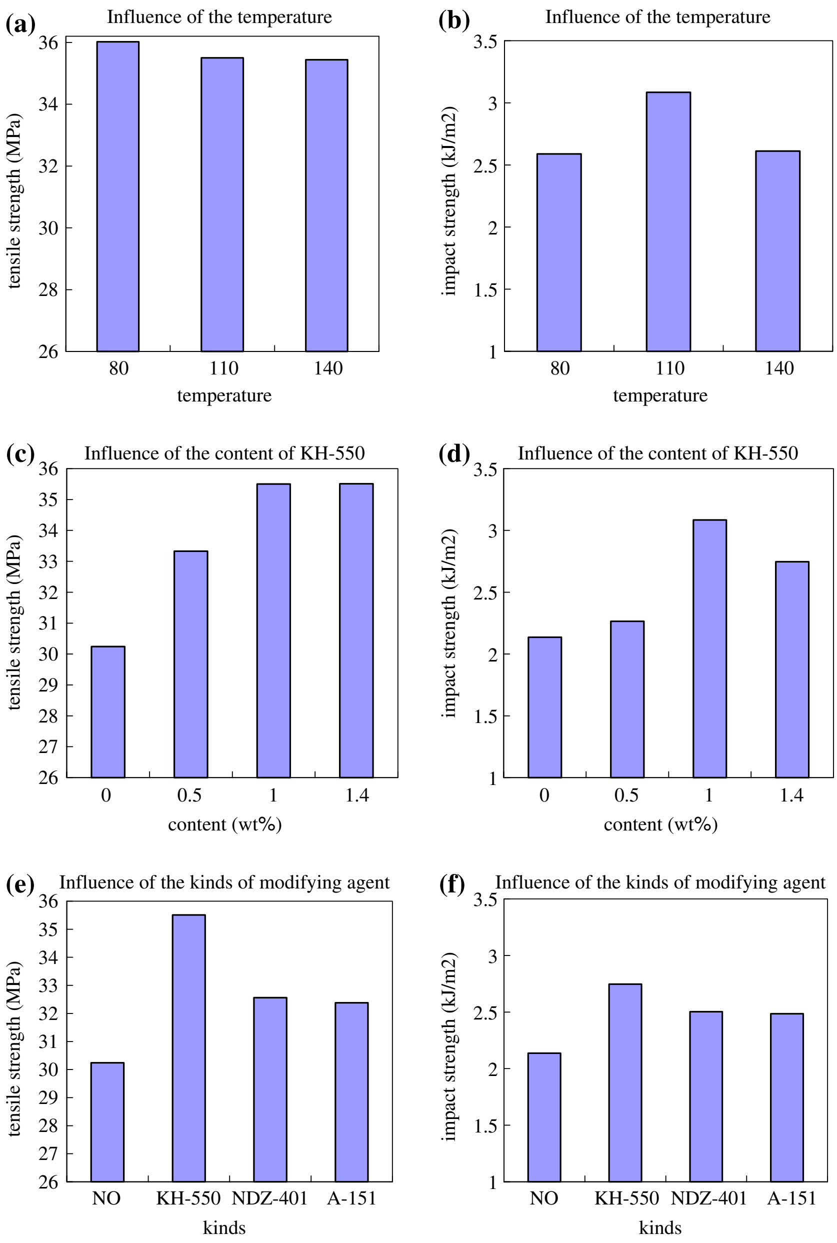


Fig. 8 Influence of temperature, content and kinds of modifying agent on the mechanical properties of silica filled PP composites, the filler content is 20%. (**a**) Influence of the temperature on tensile strength, (**b**) Influence of the temperature on impact strength, (**c**) Influence of the content of KH-550 on tensile strength, (**d**) Influence of the content of KH-550 on impact strength, (**e**) Influence of the kinds of modifying agent on tensile strength, (**f**) Influence of the kinds of modifying agent on impact strength

In Fig. 8a and b, the silica was modified by 1 wt% of KH-550, it can be seen that the modifying temperature has little effect on tensile strength, but has much effect on impact strength of PP composites, the optimal temperature is 110 °C. The influence of the content of modifying agent on the mechanical properties of PP composites are shown in Fig. 8c and d, the silica was modified by KH-550 at 110 °C. From the figures it can be seen that when the content is below 1.0 wt%, both tensile strength and impact strength increase as the content increases, and reach maximum at 1.0 wt%. The phenomenon is presumably due to improvement of the interface compatibility between PP and silica for forming a monomolecular layer of coupling agent KH-550 at 1.0 wt% on the surface of the silica particles. The KH-550 is just as a molecular bridge between the interface, which improves the miscibility and interface adhesion, enabling a more efficient stress transfer from PP matrix to silica. When the content of KH-550 is over 1.0 wt%, the excess content of coupling agent will form multilayer on the base of the first layer by Van der Waal's force. It tends to increase aggregation, to damage dispersion performance, and also to reduce the interface binding force between silica and PP matrix, which leads to the decrease of mechanical properties of PP composites.

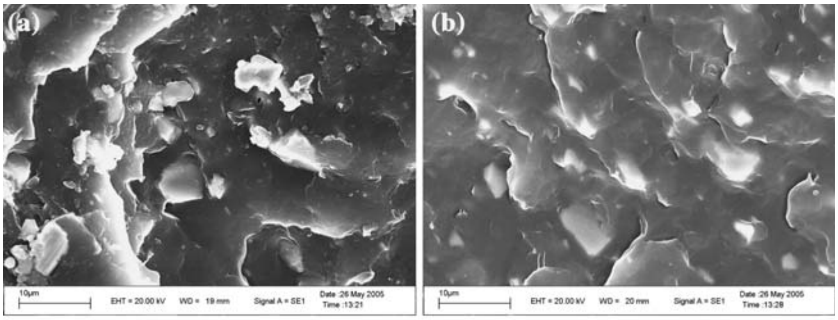
The influence of different agent on mechanical properties of PP composites are shown in Fig. 8e and f, the silica was modified by 1 wt% of agents at 110 °C. It can be seen that the PP composites filled with the modified silica have better mechanical properties than filled with the unmodified silica, and the best modifying agent is KH-550 in experiment. In former viscosity results, the silica modified by NDZ-401 has the best compatibility with paraffin, but the mechanical properties of the PP composites filled with the NDZ-401 modified silica are not the best in mechanical experiments. This phenomenon is presumably due to the magnitude of the interface bonding forces between the modified silica and the PP: the silica modified by KH-550 will form the stronger interface binding forces in the interface of composites than the silica modified by NDZ-401, which may prove the theory that the mechanical properties of the powder filled composites are not only influenced by the compatibility or miscibility between powder and matrix, but also influenced by the interface adhesion between the particles and the matrix. Thus, the viscosity of the particles-paraffin suspension can only evaluates the compatibility between the particles and paraffin, evaluates the modification performance in advance when the same modifying agent is used.

Scanning electron microscopy (SEM) was used to examine the structure of the fracture surface and the interface between particles and matrix, and to justify the variation of the measured mechanical properties.

Figure 9 shows the impact fractographs of PP composites filled with 20 wt% unmodified and modified silica. In Fig. 9a, poor unmodified silica particles dispersion and significant particles agglomeration in the PP matrix is presented. Further, the voids, which can be seen between the unmodified silica and matrix, indicate the poor interface adhesion between them. The polar nature of unmodified silica and non-polar nature of PP matrix can be cited as one of the possible causes for this observation. All these factors contribute to the poor stress transfer from matrix to silica resulting in poor mechanical properties of PP composites filled with unmodified silica. In contrast to the morphology of fracture surfaces of unmodified silica filled PP composites, the modified silica filled PP composites (Fig. 9b–d) show better compatibility between the modified silica and PP matrix. In Fig. 9b–d, the modified silica do not exhibit a tendency towards formation of large agglomerates, a significantly stronger silica PP matrix interface is evident from the micrographs, and the modified silica were well wetted with PP matrix. The above results are in reasonable agreement with the results of the mechanical properties of silica filled PP composites.

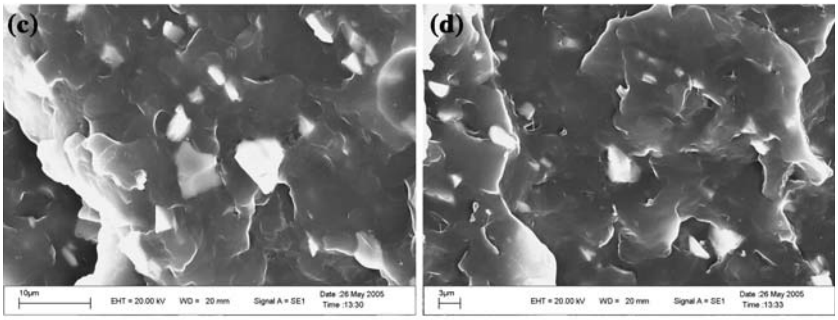
Conclusion

In this paper, a novel technology for powder dispersion and surface modification was studied by experiment, the influence of some parameters on dispersion and modification performance is studied. In the process of dispersion, the dispersion forces induced by the designed internal-swirling nozzle are larger than other nozzles



Unmodified silica filled PP

KH-550 modified silica filled PP



NDZ-401 modified silica filled PP

A-151 modified silica filled PP

Fig. 9 SEM micrographs of the impact fractographs of PP composites filled with 20 wt% unmodified and modified silica. (**a**) Unmodified silica filled PP, (**b**) KH-550 modified silica filled PP, (**c**) NDZ-401 modified silica filled PP, (**d**) A-151 modified silica filled PP and the dispersion uniformity for particles dispersed by internal-swirling nozzle is also better than other nozzles. In the processes of modification, the kinds and content of modifying agent have much effect on the performance of powder modification. For silica, used in this paper, the optimal content of KH-550 is 1 wt%, the optimal agent kinds is NDZ-401 for the compatibility between the powder and paraffin, but for mechanical properties of PP composites the optimal agent kinds is KH-550, this phenomenon is presumably due to that the mechanical properties of the powder filled composites are not only influenced by the compatibility or miscibility between powder and matrix, but also influenced by the interface adhesion between the particles and the matrix: the silica modified by KH-550 will form the stronger bonding forces in the interface of composites than the silica modified by NDZ-401. The results show that the developed technology is effective for powder dispersion and surface modification.

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