



## Original Article

### Insight into in-situ modification of white carbon black with surfactant/silane coupling agent via surface grafting and its performance

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## ABSTRACT

Hydrophobic and highly dispersed modified white carbon black particles were prepared using surfactant sodium dodecyl sulfate as a modifier and the vanadium-extraction residue as raw material. This study investigated the effects of modification temperature, pH value, aging time and dosage of modifier. In order to reflect the advantages of surfactant sodium dodecyl sulfate modification, the silane coupling agent  $\gamma$ -methacryloxypropyl trimethoxysilane was used to compare with it. The modified white carbon black prepared by in-situ grafting method with sodium dodecyl sulfate and  $\gamma$ -methacryloxypropyl trimethoxysilane was compared. X-ray diffraction and fourier transform infrared spectrometer results indicate that the modified product has an amorphous structure and the modifier exists on the surface of white carbon black in the form of chemical grafting. The modified product is added as fillers to rubber and the mechanical properties of rubber show that the white carbon black modified by sodium dodecyl sulfate has a lower modulus of elasticity, a higher maximum tensile stress and the strain at yield. The results of scanning electron microscope images of rubber and particle size of modified white carbon black demonstrate that the fine-grained modified white carbon black particles are more likely to form a network structure with the rubber to enhance the mechanical properties of the rubber.

**Keywords:** Vanadium-extraction residue; Modified white carbon black; Surface modification; Rubber; High mechanical properties

## INTRODUCTION

White carbon black (WCB) is an amorphous silica with porous structure. It possesses many outstanding properties, including high melting point, low density, high specific surface area, good thermal and stability dispersibility (Salavati-Niasari et al., 2013; Wang et al., 2019; Landarani-Isfahani et al., 2014; Salimi et al., 2009), which makes it become versatile materials and can be used as as catalysts support, pigments, chromatographic adsorbent, chemo-mechanical polishing, hybrid-composite materials, humidity sensors, thermal insulators etc. Especially in rubber industry, WCB has gradually replaced carbon black as fillers in

rubber compounding process to enhance the mechanical properties of product (Seliem et al., 2013; Sáenz et al., 2003; Murniati et al., 2020). However, WCB generally presents strong hydrophilicity due to abundant silanol groups on the surface and cannot form good network structure with rubber molecules, which leads to the poor reinforcement effect on rubber (Gauthier et al., 2004; Wang et al., 2020a). Therefore, surface modification of WCB are commonly necessary to enhance its compatibility with rubber before it is used as fillers in rubber industry (Sarkawi et al., 2013, 2014; Jesionowski et al., 2003).

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Presently, wet modification method is widely adopted to obtain the modified WCB where WCB is mixed with modifier in organic solvent followed by heating, boiling, refluxing, separating, and drying and the modifiers used mainly include organohalogenated silanes, silane coupling agents, siloxanes, and alcohol compounds (Itzhaik-Alkotzer et al., 2019; Marczak et al., 2016). The most widely used modifier for WCB is silane coupling agents. The silanol groups on the surface of WCB can react well with the modifier to form a chemical bond so that the modifier is adsorbed on the surface of WCB to complete the surface modification process (Bhagat and Rao, 2006; Fujino et al., 1992; Bareiro and Santos, 2014). In view of the above modification process, it can be seen that this method is not only complex and time-consuming but also causes threat to the environment due to the use of organic solvent (Ren et al., 2016; Ma et al., 2010). In order to shorten the preparation process of modified WCB, in-situ grafting modification method was proposed (Song et al., 2014). The improved method is adding tetraethyl orthosilicate (TEOS) and modifiers to organic solvent, so that TEOS can complete modification process while generating WCB. However, this method is a reaction between organic substances, which will cause problems such as organic chemical pollution and long reaction time. In addition, sodium silicate is used to prepare WCB through chemical precipitation in industry. And the modifier is insoluble in water and cannot be in good contact with silicon-rich solution, resulting in poor modification effect and low modification efficiency. Hence, water-soluble surfactants are considered to modify WCB. Sodium dodecyl sulfate (SDS), a kind of surfactant, is widely used in the powder modification process. He et al. (He et al., 2019) used SDS as modifier to increase the hydrophobicity of  $KNO_3$  to achieve the anti-caking effect. Yu et al. (2019) used SDS to modify the spray-dried nano-silica and prepared hydrophobic nano-silica. Consequently, SDS

can significantly improve the hydrophobic properties of the modified substance, so it is considered to modify WCB in this work.

In this study, silicon-rich solution was prepared using vanadium extraction residue (VER) as raw material and then modified WCB were prepared using surfactant as modifier with in-situ grafting methods. In order to characterize modification effect, SDS and silane coupling agent were used as modifiers to prepare WCB for comparison. In addition, the two kinds of modified products mentioned above were added to the rubber mixing process as fillers, and the properties of rubber were characterized to further reflect the quality of the modified WCB.

## MATERIALS AND METHODS

### Materials

Silicon raw material VER used in this study was obtained from sodium salt roasting-water leaching technique for vanadium extraction. The chemical compositions were shown in Table 1 and XRD pattern was shown in Fig. 1. The chemicals used in this study were sodium hydroxide, sulfuric acid, SDS (Supplied by Sinopharm Chemical Reagent Co., Ltd),  $\gamma$ -methacryloxypropyl trimethoxysilane (KH570) (Supplied by Tianjing Tianli Chemical Reagent Co., Ltd), isoprene rubber (IR), zinc oxide, sulfur, N, N-2 dicyclohexyl-2, 2-dibenzothiazole sulfonamide (DZ), 2, 2, 4-trimethyl-1, 2-dihydroquinoline polymer

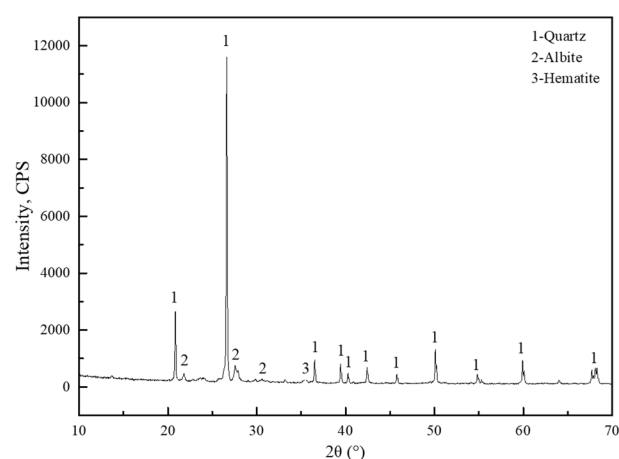


Fig. 1. XRD analysis patterns of the VER

Table 1. Main chemical composition of VER (wt %)

Element	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	P <sub>2</sub> O <sub>5</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	V <sub>2</sub> O <sub>5</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	Loss
Content/%	83.47	5.09	0.95	0.65	1.20	3.20	0.66	0.23	2.40	0.21	1.25

(RD) and stearic acid (SA). The water used in this experiment was laboratory-made ultrapure water.

### Preparation of sodium silicate from VER

According to our previous work (Wang et al., 2020b), 30 g VER powders was mixed 30 g sodium hydroxide particles to prepare silicon-rich solution at 194°C for around 2 h with stirring. The chemical reaction is shown as follows:



The leachate was filtered by a vacuum suction filter to obtain silicon-rich solution, and then the concentration of SiO<sub>2</sub> was measured. Finally, silicon-rich solution was diluted to a solution with SiO<sub>2</sub> concentration of 20 g/L in order to control the time of gel formation.

### Preparation of modification WCB

Chemical precipitation process was used to prepare modified WCB product and the silicon-rich solution was used as precursor. Different from other modified WCB preparation methods, the modifier is first added to the precursor solution. For gelation of the solution, sulfuric acid was added to adjust pH value. After gelation, the gel was filtered by a vacuum suction filter and washed several times to remove by-product Na<sub>2</sub>SO<sub>4</sub>. Finally, put the washed filter cake into a vacuum drying oven at 50°C to obtain a modified WCB powder. In this study, two different types of modifiers (SDS and KH570) were used in modification process, and the modified products obtained were WCB/SDS and WCB/KH570, respectively.

### Rubber compounding

The modified WCB was added to the rubber mixing process as fillers. The rubber compounding process is divided into two parts: rubber mastication and rubber mixing. Weigh 100 g IR into open rubber mixer, adjust roll distance to 0.8 mm, and repeat 5 times to obtain masticated rubber. Adjust roller pitch to 0.6 mm, put the masticated rubber into the open rubber mixer to wrap rollers, and then add SA, ZnO, RD, DZ and WCB in sequence. Finally,

sulfur was added, and then the rubber product is obtained after 5 passes. During the experiment, the weight ratio of rubber and compounding agent was controlled as IR: ZnO: SA: RD: DZ: S: WCB=100: 5: 2: 1: 1.5: 2: 1.

### Methods of characterization

The chemical composition of VER was analyzed with an X-ray fluorescence spectrometer (XRF, PANalytical B.V, Netherlands). The phase composition of VER and modified WCB product was analyzed by X-ray Diffractometer (XRD, Bruker-D8, Germany) working on 40 kV instrument using a Cu Kα Ni-filtered radiation (λ=1.5406 Å). Fourier transform infrared spectrometer (FTIR, VERTEX-70, Bruker, Germany) with a scanning wavenumber ranging 400 cm<sup>-1</sup> to 4000 cm<sup>-1</sup> was used to acquire infrared spectrometer of the samples. Laser particle size analyzer (BT-9300, China) was used to measure the particle size of the sample. The microscopic structure and elemental distribution of the sample were observed and analyzed using a scanning electron microscopy (SEM, JSM-IT300, JEOL, Japan), which is equipped with an energy dispersive spectrometer (EDS, Oxford, UK). The computer-controlled electro-hydraulic servo universal testing machine (SANS, CMT4304) is used to test the mechanical properties of rubber.

The properties of modified WCB are characterized by dibutyl phthalate (DBP) oil adsorption value. The method of DBP oil adsorption value test is as follows: The principle is to drop the DBP oil with a burette (precisely) onto a certain amount of WCB sample at a certain speed, and use glass rod to continuously stir and roll on the glass plate to make the mixture change from free-flowing powder to semi-plastic agglomerate. Roll WCB powder onto the glass rod, if there is no oil stain on it, the end point is judged. This operation process is required to be completed within 4 min to 6 min.

## RESULTS AND DISCUSSION

### Effect of modification parameters

#### *Effect of temperature*

As is displayed in Fig. 2, the DBP oil adsorption value was estimated under different temperature

range. It increases first and then decreases with the increase of temperature, and achieves the maximal value at 70°C. This is due to that the greater interaction between the hydroxyl groups could have with SDS and KH570 at the higher temperature and the more modifier grafted on the WCB surface. However, the dispersal state of WCB particles seems not to improve when the temperature exceeds 70°C. This can be explained that the more modifier molecules came into being the micelles in water and less of them could interact with the hydroxyl groups on WCB surface.

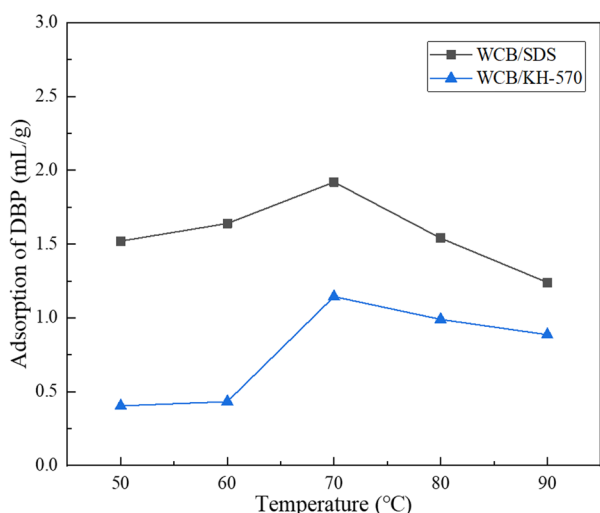


Fig. 2. Effects of temperature on the adsorption of DBP

### Effect of dosage of modifier

The effects of dosage of modifier on DBP adsorption were investigated by varying the dosage ranging from 1% to 10%. Fig. 3 shows that the DBP adsorption of WCB/SDS increases with the increase of the amount of the modifier,

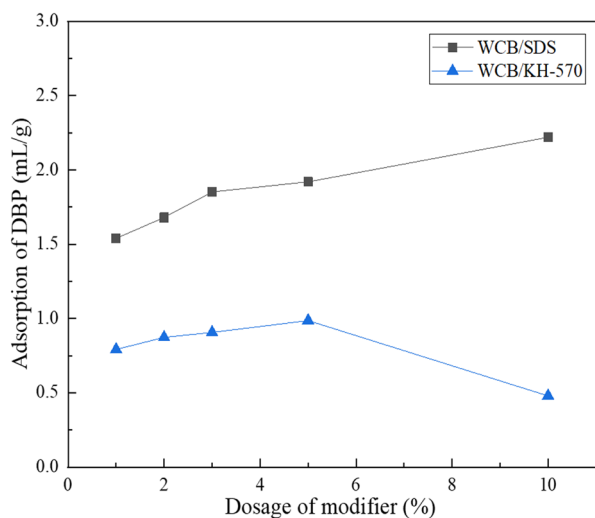


Fig. 3. Effects of dosage of modifier on the adsorption of DBP

and reaches the maximum when the amount is 10%. The DBP adsorption of WCB/KH570 increase first and then decrease, and reaches the maximum when the modifier dosage is 5%.

### Effect of pH value

The DBP adsorption of the modified product first increases and then decreases with the increase of the pH value, and reaches the maximum value at the pH value of 8, as shown in Fig. 4. The reason for this phenomenon is that the suitable pH value during the preparation of WCB is 8. When the pH value is greater than 8, the amount of WCB produced is less, and when the pH value is less than 8, the produced WCB will be dissolved by sulfuric acid.

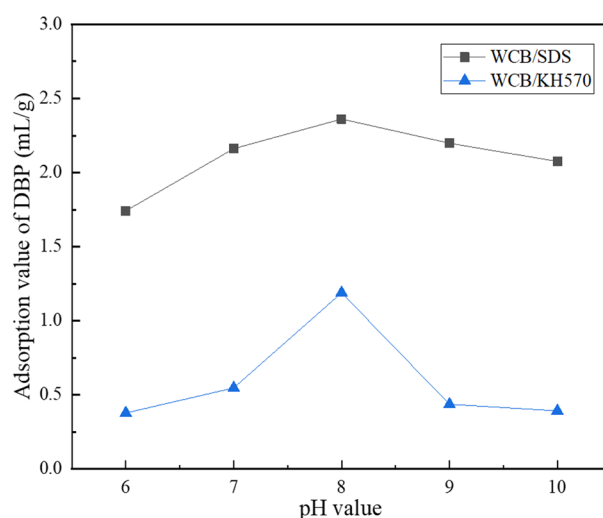


Fig. 4. Effects of pH value on the adsorption of DBP

### Effect of aging time

Fig. 5 presents the effect of aging time on DBP adsorption. The DBP adsorption of the

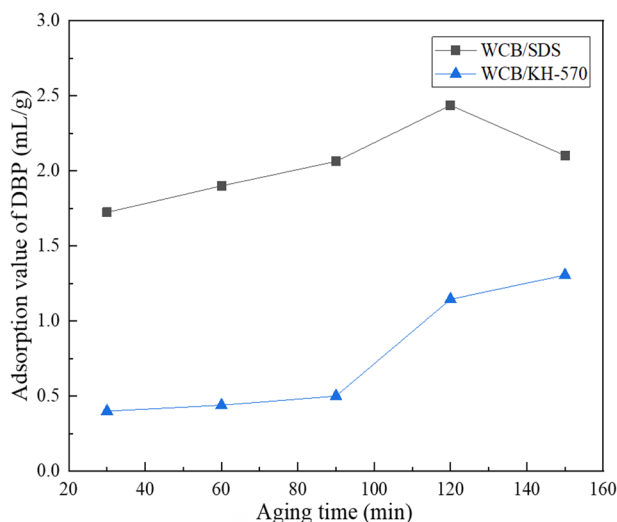


Fig. 5. Effects of aging time on the adsorption of DBP

WCB product modified by KH570 increases with the increase of aging time, and reaches the maximum when the time is 150 min. The DBP adsorption of SDS increases first and then decreases, and reaches the maximum when the time is 120 min.

It is noticed that there is a significant difference between the two kinds of modifier (SDS and KH570). Obviously, the DBP adsorption of SDS modified products is higher than that of KH570. The reason for this phenomenon is that the raw material used in the modification experiment is sodium silicate solution. KH570 is an oily silane coupling agent, which is insoluble in water. Accordingly, the modified product has excellent hydrophobicity, and it is difficult to form a structure in the solution, resulting in a smaller DBP adsorption.

### Characterizations of the modified WCB Crystal structure

The XRD patterns of precipitated WCB are shown in Fig. 6. The typical dispersion peak at 20°~25° appears in XRD patterns of modified WCB products, which demonstrates that precipitated WCB is amorphous and surface modification does not change the phase structure of precipitated WCB (Chen et al., 1997; Amin et al., 2016).

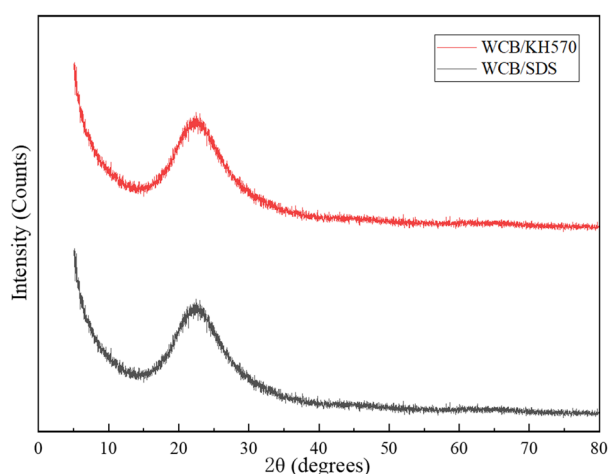


Fig. 6. XRD pattern of modified WCB products

### The modifiers grafting on the surface of WCB.

In order to estimate the mechanism of modifier and WCB, the FT-IR spectrums of modified products are shown in Fig. 7.

The adsorption peaks at 799, 791 1097, 1063, and 1137 cm<sup>-1</sup> are corresponded to the bending

vibration, symmetric stretching vibration, asymmetric stretching vibration of Si-O-Si (Garbassi et al., 1995; Kim et al., 2009; Tai et al., 2008). The band at 767 and 940 cm<sup>-1</sup> are assigned to the Si-OH deformation vibration (Cui et al., 2015; Shahnani et al., 2018). From curves (a and b) in Fig. 7, the typical stretching vibrations of the C-H are found at 2846 and 2914 cm<sup>-1</sup>, which results from the -CH<sub>2</sub> and -CH<sub>3</sub> in the modifier, respectively. The adsorption peaks at 1638, 3455, and 3417 cm<sup>-1</sup> correspond to bending and stretching vibration of -OH (Lin et al., 2012). At the same time, the adsorption peaks for 1719 cm<sup>-1</sup> correspond to the stretching vibration of C=O (Chen et al., 2017), which demonstrates that KH570 is grafted on WCB surface. Moreover, the physical adsorbed modifier and reaction byproducts of precipitated WCB were washed with ethanol for three times in surface modification process, so the physical adsorption between precipitated WCB and modifier can be eliminated. To sum up, SDS and KH570 were mainly grafted to the WCB surface by chemical bond rather than physical adsorption.

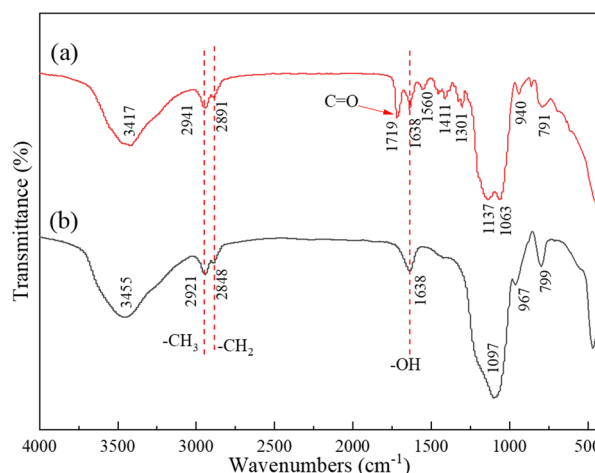


Fig. 7. FTIR spectra of modified WCB: (a) WCB/KH570, (b) WCB/SDS

### Degree of modification reaction

Fig. 8 shows the TGA plots of WCB/SDS and WCB/KH570. The samples were dried at 105°C for 4 h to remove the redundant water on the surface. However, the obvious weight loss in samples could be found in the plots. In the low temperature (<300°C), the weight loss is about 7.07 wt.%, as shown in Fig. 8(a), which could be derived from the removal of the water absorbed on the surface of modified WCB particles (Ma et al., 2010). And there is still about 5.84 wt.%



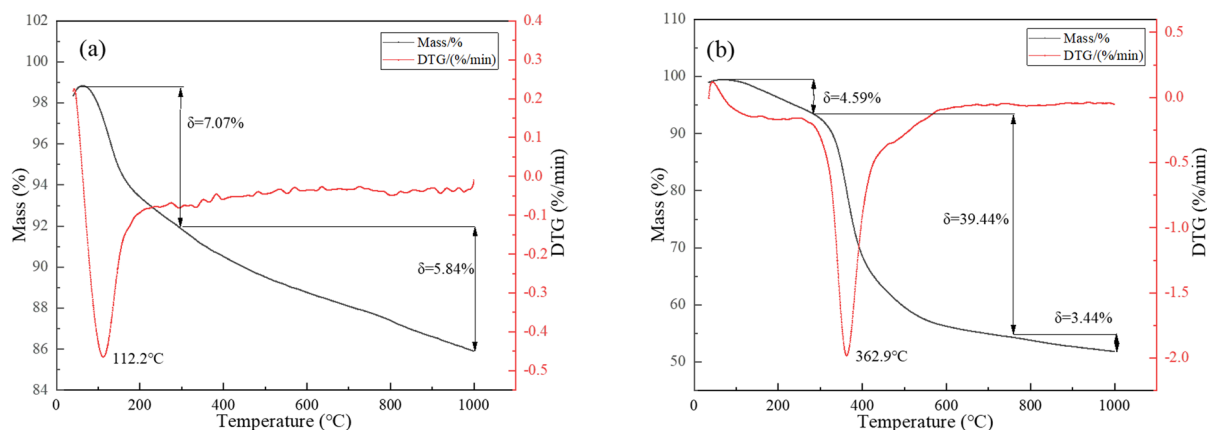


Fig. 8. TGA and DTG plots of modified WCB particles: (a) WCB/SDS, (b) WCB/KH570, (b) WCB/SDS

weight loss when the temperature is raised from 300°C to 1000°C. The weight loss is derived from the dehydration condensation of Si-OH in WCB molecules. In addition, the weight loss of KH570 modified WCB is less than that of WCB/SDS in the plots till the temperature reaches 300°C in Fig. 8(b). This indicates that the modification process made less water to be absorbed on the modified WCB surface. This result is in good agreement with the hydrophobicity of the modifiers (KH570>SDS). And there is about 39.44 wt.% weight loss when the temperature was raised from 300-800°C, the weight loss is caused by Si-OH dehydration and condensation, where Si-OH comes from the incomplete reaction in the WCB modification process.

**Particle size analysis of WCB**

The results of the particle size and micro morphology of modified WCB are shown in Fig. 9.

The median particle size ( $D_{50}$ ) of precipitated WCB is 15.65  $\mu\text{m}$  and 29.24  $\mu\text{m}$  for WCB/

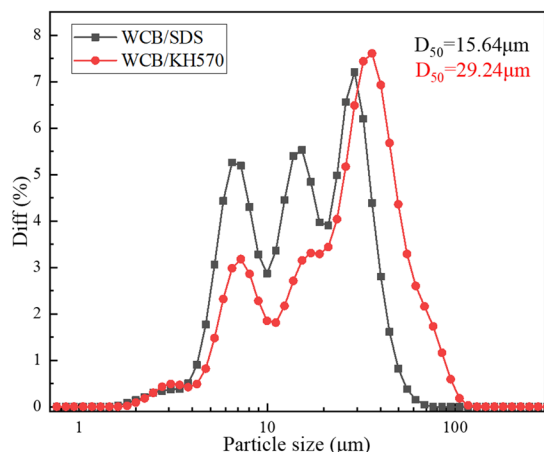


Fig. 9. Particle size of modified products

SDS and WCB/KH570, respectively. The  $D_{50}$  of precipitated WCB decreases after surface modification. The modifier could adsorb on the surface of WCB particles, which form a mesh structure covered the particle surface and prevent the particle gathering larger. From the results, the anti-agglomeration effect of SDS is more significant. Compared with the WCB/KH570, the particle size distribution of WCB/SDS is more at the fine particle level, and the particle size distribution of the modified product obtained by in-situ grafting with SDS is more uniform.

**Characterizations of the rubber**

**Characterization of mechanical properties of rubber**

The stress-strain curve of rubber is shown in Fig. 10. It indicates that SDS as a modifier has a better strengthening effect on the mechanical properties of rubber compared with KH570.

The specific mechanical performance data is shown in Table 2.

We can see that the mechanical properties of

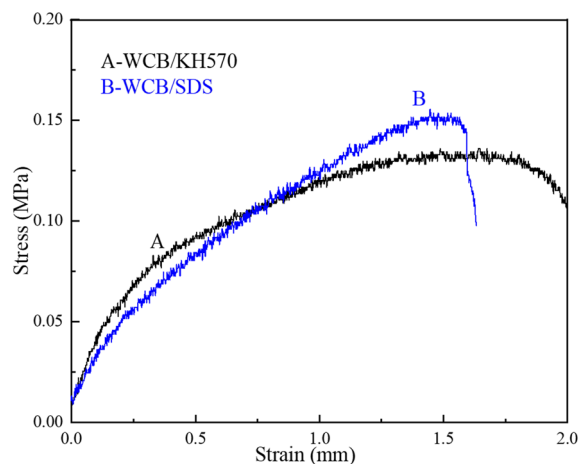
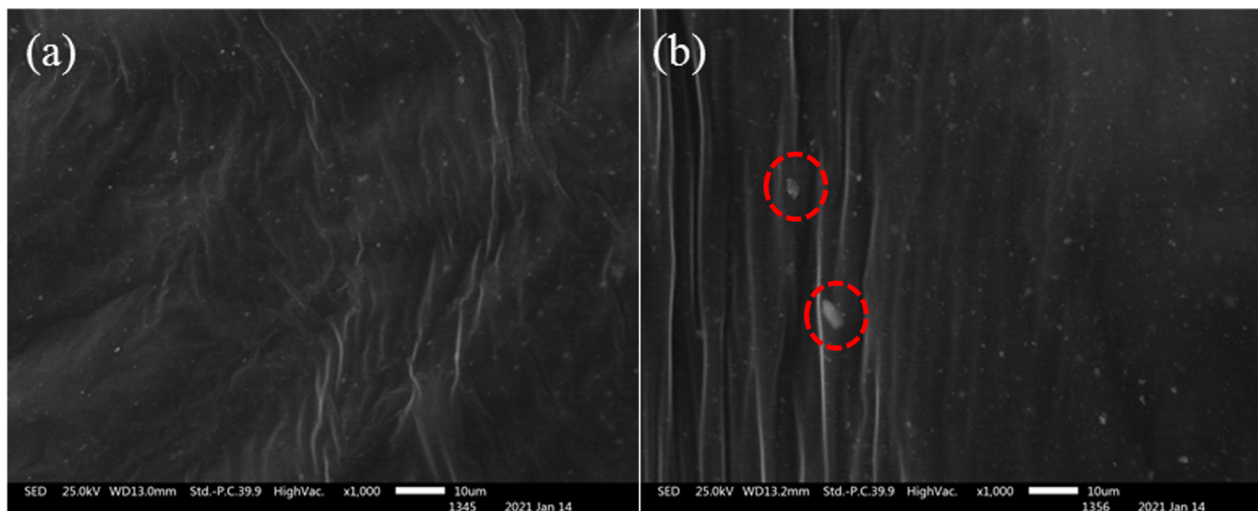


Fig. 10. The strain-stress curve of rubber

**Table 2.** The results of mechanical performance test

Sample	Elongation at break/%	Elastic Modulus/MPa	Maximum force/MPa	Tensile Strength/%	Strain at yield/%	Strain at tensile strength/%
A	202.7	0.36	8.16	0.14	6.19	145.72
B	187.46	0.26	11.25	0.22	12.94	167.86



**Fig. 11.** Distribution of modified WCB particles in rubber: (a) WCB/SDS, (b) WCB/KH570

sample B (WCB/SDS) have more advantages than sample A (WCB/KH570) from the graph above. The elastic modulus of the rubber product added with the modifier was significantly reduced, indicating that the addition of modified WCB can effectively reduce the elasticity of the rubber and enhance the plasticity of the rubber. At the same time, the maximum tensile force that the rubber can withstand after the addition of modified WCB is also greatly increased, indicating that the modification can increase the strength of the rubber.

#### ***Analysis of the microscopic appearance of rubber***

The distribution of modified WCB particles in rubber is shown in Fig. 11. As is shown in Fig. 11(a), there are a large number of white particle aggregates on the rubber section, and they are evenly distributed on the section layer, indicating that the modified WCB particles are well dispersed in the rubber. So, it is easy to form a network structure with the rubber molecules to strengthen the mechanical properties of rubber. However, part of the WCB/KH570 modified WCB aggregates in rubber, as shown in Fig. 11 (b), which weakens the combination of WCB particles and rubber molecules.

#### **CONCLUSION**

In this study, SDS and KH570 were used to modified WCB with in-situ grafting method. The experiments indicate that temperature of 70°C, modifier dosage of 10%, pH value of 8, and aging time of 120 min are the optimal conditions for modification of SDS. And the optimal conditions for modification of KH570 are temperature of 70°C, modifier dosage of 5%, pH value of 8, and aging time of 150 min. Under the same conditions, the DBP oil adsorption value of WCB/SDS is higher than that of WCB/KH570.

XRD and FTIR results indicate that the modified WCB is amorphous, and the modifier is grafted onto the surface of WCB particles in the form of chemical bonds. TG analysis indicates that: compared with WCB/SDS, WCB/KH570 contains a lot of unreacted Si-OH on the surface, which demonstrates that KH570 as a modifier has a slow reaction speed and a long modification time. The modified WCB was mixed with natural rubber as fillers to prepare rubber compound. The rubber added with WCB/SDS has more excellent mechanical properties, which are mainly reflected in the reduction of the elastic modulus and the increase of the maximum force and the strain at yield.

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### ACKNOWLEDGEMENTS

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