

Preparation of Nano-silica Fume Coating Rubber Composite Particle and Its Application in Toughening the Oil Well Cement Stone

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Abstract. A kind of nano-silica fume coating rubber composite particles was prepared by the Particle Composite System (PCS), and the adhesion performance of composite particles was tested by the high-speed agitating test, the composite particles were filled into the oil well cement slurry to toughen the oil well cement stone. The results indicated that the surface of the rubber particles is coated with nano-silica layer, and the composite particles are grey; the adhesion between the silica fume and the rubber particles is good; the elastic modulus of oil well cement stone reduces by 20% and the Possion ratio increases by 5.1% as the filler fraction is 4 wt.%. Therefore, the nano-silica fume coating rubber composite particles can improve the toughness of oil well cement stone.

Introduction

The tough materials including fiber [1][2], latex particles [3], rubber particles [4] and organic resin [5] are used in the oil well cement slurry to improve the toughness and cementation performance of oil well cement stone. The latex particles and organic resin materials are limited use for their high price, the rubber particles and silica fume are widely used in toughening the oil well cement stone for the low price and wide resource. But, the density of the silica fume is heavier than that of the oil well cement; otherwise, the density of the rubber particle is much lighter than that of the oil well cement. Therefore, the rubber particles and silica fume cannot mix with oil well cement slurry evenly. To resolve the problem, the nano-silica fume is coated orderly on the surface of the rubber particles by the PCS, and the nano-silica coating rubber composite particles are prepared. The density of the nano-silica coating rubber composite particles reduces the density difference in the slurry and can be dispersed orderly, so as to improve the stability of cement slurry.

Experimental

Particles Composite System. High speed pneumatic impact particle composite technology is widely researched in all over the world [6-11]. In china, the Powder Engineering Laboratory of Materials Science and Technology of Tsinghua University is the first research institution which developed the high speed pneumatic impact PCS [12]. Now, the PCS is widely used to prepare the composite particles and finish the shape of the powder.

The components of PCS include the main mechanic, the catcher and control system. The nano-silica fume and rubber particles mixed evenly are fed into the main mechanic by the quantitative metering system. The main mechanic includes rotors, stators and the loop, as the Fig.1 shows:

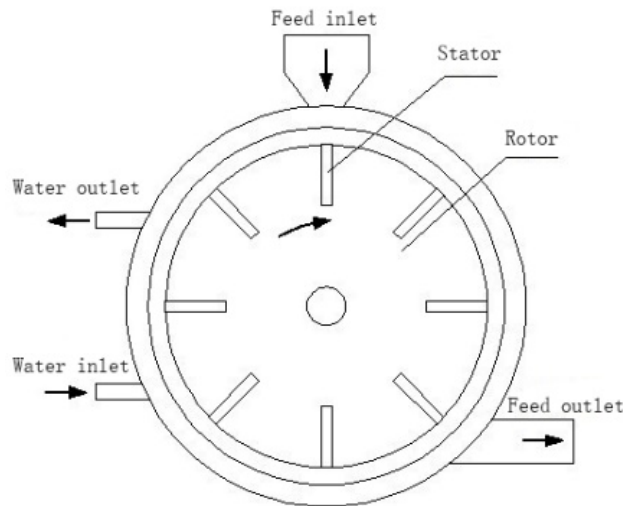
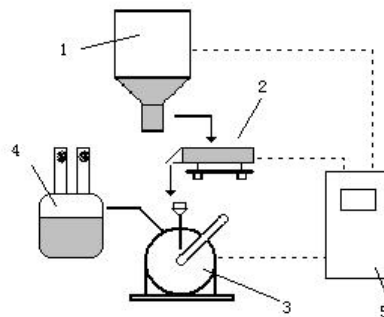


Fig.1 Schematic diagram of the main mechanic of PCS

The particle materials are dispersed by the components of the main mechanic. At the same time, the strong impact force, compressive force, frictional force and shear force among the particles can coat the small particles on the surface of the big particles or mix different particles evenly in a short time.

Composite Technic of Micron-Nano-Sized Particles Composite system. The rubber particles and the silica fume nano-sized particles with a certain proportion are fed into the PCS. The experimental parameters are adjusted, and the two kinds of particles are contacted and mixed fully to form a core-shell structure, silica fume coating rubber composite particle. The process flow as Fig.2 shows:



1.OM mixer 2.quantitative metering system 3. PCS main mechanic 4. collecting device 5. control system

Fig.2 the process flow of PCS

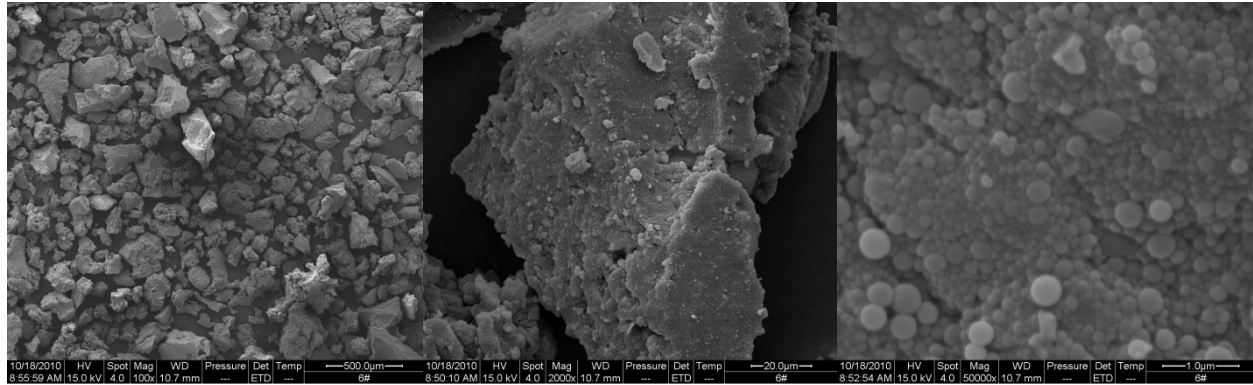
Experiment of Nano-Silica Fume Coating Rubber Particle. The nano-silica fume, with diameter range from 70nm to 500nm, was provided by the Elkem ASA Corporate Headquarters (Tianjin, China), the rubber particles were provided by the Dongguan Liyuan Rubber Products Co., LTD (Dongguan, China). The nano-silica could be coated on the surface of rubber particle orderly after being disposed by the PCS. Thus, the properties of oil well cement can be improved at the low filler content. The experimental parameters are as the table 1 show:

Table 1 Experimental parameters for nano-silica fume coating the rubber particles

Weight of rubber particles (Kg)	The content of nano-silica fume (%)	The experimental parameters of PCS	
		Rotor rotational speed(rpm)	Processing time(min)
1	5	2500	30
1	10	2500	30

Results and Discussion

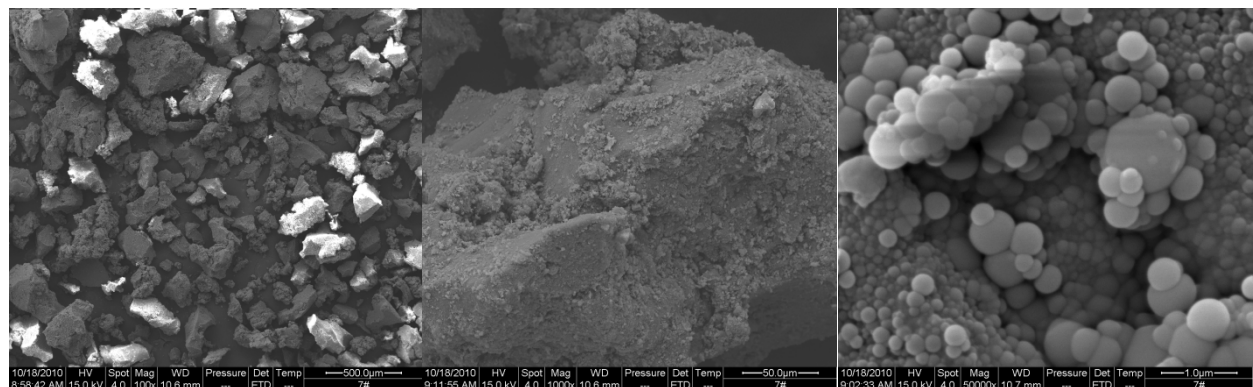
SEM of Nano-Silica Fume Coating Rubber Composite particles. The SEMs of nano-silica fume coating rubber composite particles were shown in Fig.3 and Fig.4. Fig.3 showed the composite particles with the content of nano-silica fume 5wt.%, the composite particles with the content of nano-silica fume 10wt.% was shown in Fig.4.



(a) (b) (c)

(a)100× (b) 2000× (c) 50000×

Fig.3 the SEMs of the composite particles with the content of nano-silica fume 5wt.%



(a) (b) (c)

(a)100× (b) 1000× (c) 50000×

Fig.4 the SEMs of the composite particles with the content of nano-silica fume 10wt.%

It can be seen from Fig.3 that the nano-silica fume can not be found on the surface of the rubber particle at the relatively small magnification 100 (Fig.3(a)), and the surface of the composite particle is black; when the magnification was 2000 (Fig.3(b)), the silica fume can be seen on the surface of the rubber particle, and surface of the composite particle is black gray, when the magnification is 50000 (Fig.3(c)), one can see the nano-size spherical silica particles connect together, and the surface is the light black gray. We can see from Fig.4 that the nano-silica fume cannot be found on the surface of the rubber particle at the relatively small magnification 100 (Fig.4(a)) also, and the surface of the composite particle is black; when the magnification is 1000 (Fig.4(b)), the silica fume can be seen on the surface of the rubber particle, and surface of the composite particle is off-white; when the magnification is 50000 (Fig.4(c)), the nano-size spherical silica particles connecting together can be seen, and the surface of the composite particle is light off-white. by analyzing the Fig.3 and Fig.4 comparatively, we can find that the composite particles with the content of nano-silica fume 10wt.% is whiter than those with the content of nano-silica fume 5wt.%.

The Check on the Adhesion Property of the Composite Particles. The composite particles with the content of the nano-silica fume 5wt.% were agitated at high speed. Then, one small amount of composite particles after agitation were observed by SEM, the SEM image is shown in Fig.5; another small amount of composite particles were maintained at the atmospheric pressure and with

the temperature 95°C, and then observed by SEM, the SEM image is shown in Fig.6.

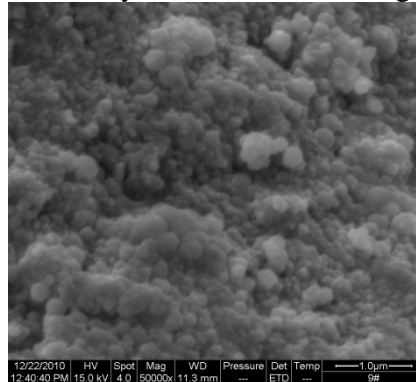


Fig.5 SEM of composite particles after high speed agitating test

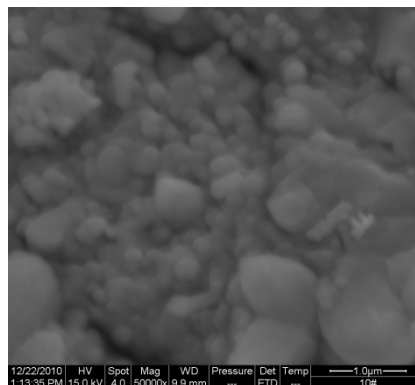


Fig.6 SEM of composite particles after high speed agitating test and maintenance at the temperature 95°C

From Fig.5 and 6, we can estimate that adhesion between the nano-silica particles and the rubber particles is good. that is, the nano-silica particles do not come off the rubber particles.

Evaluation Test for Composite Particles Toughening the Oil Well Cement. The comprehensive mechanical properties of oil well cement filled with nano-silica fume coating rubber composite particles are show in Table 2.

Table 2 Comprehensive mechanical properties of oil well cement filled with composite particles

The filling fraction of the composite particles	Possion ratio	Elastic modulus	Compressive strength
4 wt.%	0.41	13.8 GPa	23 MPa
0wt.%	0.39	17.3 GPa	31 MPa
Results comparison	+5.1%	-20.2%	-25.8%

It can be seen from Table 2 that the passion ratio of the oil well cement stone increases to 0.41 after being filled with 4 wt.%, 5.1% increased; the elastic modulus decreased from 17.3GPa to 13.8GPa, 20.2% decreased. That is to say, the composite particles can toughen the oil well cement stone.

Conclusions

A new kind of composite particles were prepared in this work, and the adhesion performance of composite particles was tested. At last, the composite particles were filled into the oil well cement slurry to toughen the oil well cement. The following conclusions were gained that the nano-silica fume are coating on the surface of the rubber particle, and the composite particles are grey; the adhesion between the silica fume and the rubber is good; the toughness of oil well cement stone is improved by filling the nano-silica fume coating rubber particles.

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