



NOTE

Study on Temperature and Time Affecting the Dispersity of Nano-Silica in Organic Solvents

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Surface modification improving the dispersity of the prepared nano-silica in organic solvents using different temperature and time were investigated. The result suggested that the optimum modification condition was that temperature was 80 °C and time was 1.5 h. The dibutyl phthalate absorption number of the optimum modified nano-silica was 3.5 cm³/g.

Key Words: Surface modification, Dispersity, Nano-silica, Temperture, Time.

Improving the dispersity of nano-silica in organic solvents had caused intensively interesting. In previous works, many researchers investigated surface modification improving the dispersity of nano-silica in organic solvents using titanate, oleic acid vinyltriethoxysilane and aminopropyl triethoxysilane^{1,2}. These modification effects were not ideal. Therefore silane coupling agent as a new modification agent was considered and investigated^{3,4}. Li *et al.*⁵ and Zhou *et al.*⁶ investigated surface modification improving the dispersity of nano-silica in organic solvents using 0.05 mol/L-methacryloxypropyltrimethoxy silane (MOPMS0KH570) anhydrous alcohol solution. Li *et al.*¹ investigated the concentration and amount of KH-570 on modified effect.

Temperature and time were two important factors affecting the modification effect. There were no reports on the effect of temperature and time on improving the dispersity of nano-silica in organic solvents. The objective of this study was to gain an optimum temperature and time of improving the dispersity of nano-silica in organic solvents. The dibutyl phthalate (DBP) absorption number was used as the index of evaluating the modification effect.

KH-570 was purchased from Crompton Shuguang Organo-silicon Co. (Nanjing). Nano-silica was produced from rice hull ash. All other reagents were of analytical grade and used without further treatment.

Selection of an optimum temperature and time: Temperature and time were used as the controllable factors. Their levels were listed in Table-1. An orthogonal array L₄(2³) was used to determine the optimum modification condition (Table-2).

TABLE 1
FACTORS AND LEVELS DESIGN
OF THE ORTHOGONAL EXPERIMENT

Level	Factors	
	Temperture (°C) (A)	Time amount of KH-570 water solution (h) (B)
1	75	1.0
2	80	1.5
3	85	2.0

TABLE-2
ORTHOGONAL ARRAY* (DBP ABSORPTION
NUMBER OF NANO-SILICA: 2.0 cm³/g)

Sample	A	B	DBP absorption number (cm ³ /g)
L1	1	1	2.8 ± 0.1 ^a
L2	1	2	3.1 ± 0.1 ^a
L3	1	3	2.7 ± 0.1 ^a
L4	2	1	3.1 ± 0.1 ^b
L5	2	2	3.5 ± 0.1 ^b
L6	2	3	3.1 ± 0.1 ^b
L7	3	1	2.7 ± 0.1 ^c
L8	3	2	3.1 ± 0.1 ^c
L9	3	3	2.6 ± 0.1 ^c
K ₁ (cm ³ /g)	8.6	8.6	—
K ₂ (cm ³ /g)	9.7	9.7	—
K ₃ (cm ³ /g)	8.4	8.4	—
Variance (cm ³ /g)	1.3	1.3	—

*Values are means ± SD (n = 3). Values followed by the different letter in the same column are significantly different ($p \leq 0.05$).

Thirty gram of the prepared nano-silica and 300 mL of water were added in a 500 mL flask. The mixture was heated in water bath with stirring at 250 rpm (temperature from 75 to

85 °C, time from 1 to 1.5 h, respectively). 90 mL 0.4 mol/L KH-570 water solution was added to the flask at 3 mL/min by a constant flow pump¹. The mixtures were treated by an Ultrasonic Cell Muller at 90 W for 15 min. Finally the mixtures were centrifuged at 3000 rpm for 10 min and filtered through Whatman ashless filter paper¹. The filter cakes were repeatedly washed with deionized water¹. The filter cakes were dried at 80 °C for 12 h to get different modified nano-silica samples.

According to the STANDARD (HG/T 3061-2008) of China, 1 g of the sample was dried at 105 °C for 1.5 h and placed on a glass board (170 mm × 140 mm). Dibutyl phthalate was rapidly added in the prepared sample. The mixture was stirred by a glass rod and constantly pressured until it was completely crushed. Then the dibutyl phthalate was slowly added in the crushed sample with stirring and pressuring until the sample was totally infiltrated by it. The volume of the dibutyl phthalate consumed was designated as the dibutyl phthalate absorption number of the sample.

Statistical analysis: Statistical analysis was carried out using ORIGIN 7.5 (OriginLab Inc., USA).

Modification principle of nano-silica: Silicate was produced when sodium silicate and sulfuric acid were mixed up¹⁻³. The elements Si and O in the silicate connected by a covalent bond form a defective three-dimensional structure¹⁻³. A lot of -OH groups are left on the surface of nano-silica. Therefore nano-silica is hydrophilic, easy to agglomerate and difficult to be dispersed in organic solvents¹⁻³. KH-570 is a long chain organic molecule and can be hydrolyzed to produce R-Si-OH (R, CH₂=C(CH₃)-COO-(CH₂)₃-Si-). The -OH group of the R-Si-OH group reacts with the -OH group of nano-silica when nano-silica and KH-570 are mixed together¹⁻³. The -Si-R group is connected with the surface of nano-silica. The combination produces steric hindrance, which prevents nano-silica from agglomeration¹⁻³.

Determination of optimum temperature and time:

According to the chemical balance theory, temperature and time could greatly affect the chemical balance. So temperature and time were selected as the controllable factors.

As shown in Table-2, among these modified samples, the dibutyl phthalate absorption number of the modified nano-silica was the highest (3.5 cm³/g) when temperature was 80 °C and time was 1.5 h. So the optimum modified condition was that the temperature was 80 °C and time was 1.5 h. It was due to that the modification reaction could not carry out completely when temperature was too low and there was steric hindrance on the surface of nano-silica when temperature was too high. It was due to that the modification reaction could not carry out completely when time was too short and there was steric hindrance on the surface of nano-silica when time was too long.

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