Letter

Preparation process of coal tar pitch powder and its stability research

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Abstract

The coal tar pitch powder, whose size less than 200 mesh, was prepared by cryogenic grinding technology with the high temperature coal tar pitch. The coal tar pitch was frozen at the friability temperature and then grinded with some dispersants in. The pitch powder was screened to achieve particles with size less than 200 mesh. The sodium fatty alcohol ether sulfate (AES) was the best dispersant after election. The effects of preservation time, temperature and dispersants on the stability of coal tar pitch powder were studied.

Keywords : coal tar pitch, coal tar pitch powder, stability, dispersant, AES

1. Introduction

Reserve of coal is abundant in the world, while oil and gas are relatively short. And the energy structure becomes more and more prominence. Coal is the dominant energy source of power generation, which is facing double pressures of the growing demand for energy and environmental protection. A large amount of coal tar pitch was produced during the production of Coal tar oil. The potential production of coal tar pitch is about 6000kt/a in China and 2000kt/a in Shanxi. The research and application of coal tar pitch involve many fields, such as carbon materials, electrode material, impregnating pitch, needle coke, carbon fibers, paving and building materials[1)-8)],but the efficiency of coal tar pitch's utilization is still very low in China. This problem is outstanding, so researching and expanding deep processing of coal tar pitch is extremely important. Using coal tar pitch as a fuel is an effective way of comprehensive utilization of resources. However, coal tar pitch is difficult to be burned completely resulting in environmental pollution due to its complex structure and high viscosity[9)]. Others studied that emulsified asphalt emulsion was produced with coal tar pitch and water at high temperature, and then melt to a liquid fuel after reducing temperature[10), 11)]. The main drawback of this method is that a large number of toxic and harmful gases were produced during the heating and melting of coal tar pitch and the cost of energy.

There are some similarities between coal tar pitch and coal in composition, structure and character. The hard-coal could be obtained directly by crushing and then the coal powder was prepared[12)]. However, coal tar pitch is black solid at room temperature and has no fixed melting point. It will be soften and then melt when heated, and the lower the softening point is, the easier it is to be soften. Coal tar pitch powder can not be crushed with traditional method because of the special composition and character of coal tar pitch. The coal tar pitch powder that meeting the requirements was prepared by the freeze grinding technique according to the low-temperature brittleness[13)-14)]. At present, the freeze grinding technology has been widely used in food, herbs and other industries[15)-18)]. The coal tar pitch powder prepared by freeze grinding technology can be burned directly as fuel and added to the fuel oil system, similar to the oil-coal-water slurry[19)]. It can also be used to prepare a new coal tar pitch water slurry similar to the coal water slurry [20)-22)]. In addition, coal tar pitch powder can also be used in the preparation of polymer materials[23)-24)]. Therefore, it is necessary to study the preparation of coal tar pitch powder and the stability of it.

2. Experimental

2.1 Raw material and reagents

The raw material of the coal tar pitch powder was selected in the high temperature coal tar pitch produced by distillation of coal tar from Shanxi JinYao Coking Co., LTD.. The soften point of high temperature coal tar pitch was determined by ring and ball method. The results were shown in Table 1, while the results of the elemental analysis in Table 2.

Table 1 showed that the soften point of high temperature coal tar pitch was 113.9°C. Table 2 showed that the principal component of high temperature coal tar pitch was carbon.

The brittle temperature of high temperature coal tar pitch was measured by DSC. The results showed that the brittle temperature of high temperature coal tar pitch was -8.97°C, which could be seen in Figure 1.

JL-C01 medium-setting emulsifier, JL-C02 slow-crack emulsifier, industrial products,were bought from LuoHe city TianLong chemical industry Co., LTD.; naphthalene sulfonate-formaldehyde condensates, industrial products, were purchased from Taiyuan city concrete concrete additive Co., LTD.; sodium lignosulphonate, industrial product,were purchased from Anyang city double loop assistant Co., LTD.; polyvinyl alcohol , industrial product, were purchased from Shanxi Sanwei Co., LTD.; sodium fatty alcohol ether sulfate (AES) and other reagents were national analytical pure.

Table 1 The soften point of high temperature coal tar pitch.

Testing times	1	2	3	4	5
determination results [°C]	110	110.3	116.2	117.1	115.8
Average value [°C]			113.9		

Table 2 The ultimate analysis of high temperature coal tar pitch.

Testing times	Element content [%]			
	C	N	H	O
1	92.90	0.94	4.27	1.89
2	92.63	0.97	4.24	2.16
Average value	92.76	0.96	4.26	2.02

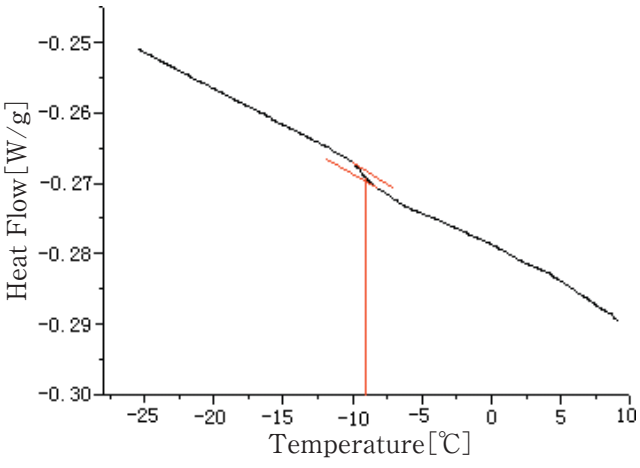


Figure 1 The DSC analysis of high temperature coal tar pitch.

2.2 Instrument and equipment

The Haier BD-100 LTB freezers are used to freeze the coal tar pitch to brittle temperature. FW-100 type high speed crusher is used to grind the coal tar pitch to powers. The softening point tester is used to measure the soft temperature of coal tar pitch. The 200 mesh standard test sieves is used to screen the coal pitch powers. The German Elementar company Varioel elemental analyzer is used to measure the content of main elements in the coal tar pitch. The U.S. TA company Q100DSC detector is used to measure the fragile temperature of the coal tar pitch. The HORIBA company LA-300 laser particle size analyzer is used to measure the particle size of the coal tar pitch. The JEOL company JSM-35C Scanning Electron Microscope is used to observe the changing of morphology of the coal tar pitch when time and temperature changed.

2.3 The preparative principle and process

Frozen grinding method was used according to low-temperature brittleness of the materials. This method's principal was that when the temperature decreased, hardness and brittleness of the materials were increased, while plastic and toughness reduced. A certain particle size of coal tar pitch powder can be prepared by frozen grinding since coal tar pitch has low-temperature brittleness. But the coal tar pitch powder with size less then 200 mesh has a large surface energy, leading to conglobation. Therefore, the dispersants would be added in the preparation process.

The coal tar pitch was frozen to become brittleness, and then dispersants were added. The high speed grinder was used to crush them, which would be screened in the final section. The coal tar pitch powder obtained which can be stored stably at room temperature.

The process of preparing coal tar pitch power with size less than 200 mesh by frozen grinding technology was as follows. First, The coal tar pitch was frozen for 24 hours below brittleness temperature. And then a high speed grinder with speed of 24000r/min was used to grind it, controlling the grinding time not to exceed 30s. Second, the coal tar pitch powder with size less than 200 mesh was obtained after being cooled and screened with 200 mesh standard sieve. The powder was stored respectively at 20°C, 30°C and 40°C for 30 or 60 days, whose stability was studied then.

3. Experimental results and discussion

3.1 The influence of dispersants on the preparation of coal tar pitch powder

The high temperature coal tar pitch powder with size less than 200 mesh was prepared with 27 kinds of dispersants in, whose dosages were one percent. The coal tar pitch powder not added with anything was prepared as a comparison at the same time. The influence of different dispersants on the stability of coal tar pitch powder was shown in Table 3.

Table 3 Effects of dispersants on preparing ≤ 200 mesh coal–tar pitch powder.

Types	Names	Percent of sifting out [%]	Types	Names	Percent of sifting out [%]
	No	86.27	Nonionic	Polyoxyethylene nonyl phenyl ether	77.34
Anion	Naphthalene sulfonate-formaldehyde condensates	90.56		Polyoxyethylene sorbitan lauric acid ester	80.09
	Sodium dodecyl benzene sulfonate	92.80		Polyoxyethylene sorbitan monopalmitate ester	79.88
	Dioctyl sodium succinate	91.22		Polyoxyethylene sorbitan monostearate ester	81.80
	Sodium dodecyl sulfate	88.59		Polyoxyethylene sorbitan monoolein ester	80.69
	Sodium decyl diphenyl ether disulfonate	95.05		Sorbitan monooleate ester	82.10
	Magnesium stearate	91.47		Polyglycol octylphenyl ether	75.25
	Sodium lignosulphonate	82.09	Amphotericionic	Dodecyl betaine	81.21
	Sodium fatty alcohol ether sulfate (AES)	95.40		Sodium laureth imidazoline acetate	93.75
Cation	JL–C01 medium–setting emulsifier	93.88	Macromolecule compound	Polyvinyl alcohol	71.69
	JL–C02 slow–crack emulsifier	78.82		Alginate sodium	82.24
	Dodecyl dimethyl benzyl ammonium chloride	92.56		Gelatin	62.35
	Cetyl trimethyl ammonium bromide	92.97	Other substance	N–methyl pyrrolidon	68.76
	Dodecyl dimethyl benzyl ammonium bromide	94.45		Diatomite	72.04

The results showed that anion and cationic dispersants were better than nonionic, amphoteric ionic and macromolecule compound. The AES was the best among the dispersants used. The dispersants whose percent of sifting out was greater than 90% were naphthalene sulfonate-formaldehyde condensates, sodium dodecyl benzene sulfonate, dioctyl sodium succinate, sodium dodecyl sulfate, dioctyl sodium succinate, sodium dodecyl sulfate, sodium decyl diphenyl ether disulfonate, magnesium stearate, JL-C01 medium-setting emulsifier, dodecyl dimethyl benzyl ammonium chloride, cetyl trimethyl ammonium bromide and sodium laureth imidazoline acetate.

On the basis of the above results, we selected the dispersants, whose percent of sifting out was greater than 90%, to prepare the high temperature coal tar pitch powder with size less than 200 mesh. The influence of the dosages of dispersants on the preparation process was investigated. The results showed that AES was the best dispersant and the optimum dosage was 1.5%.

3.2 The effect of the stability of coal tar pitch powder

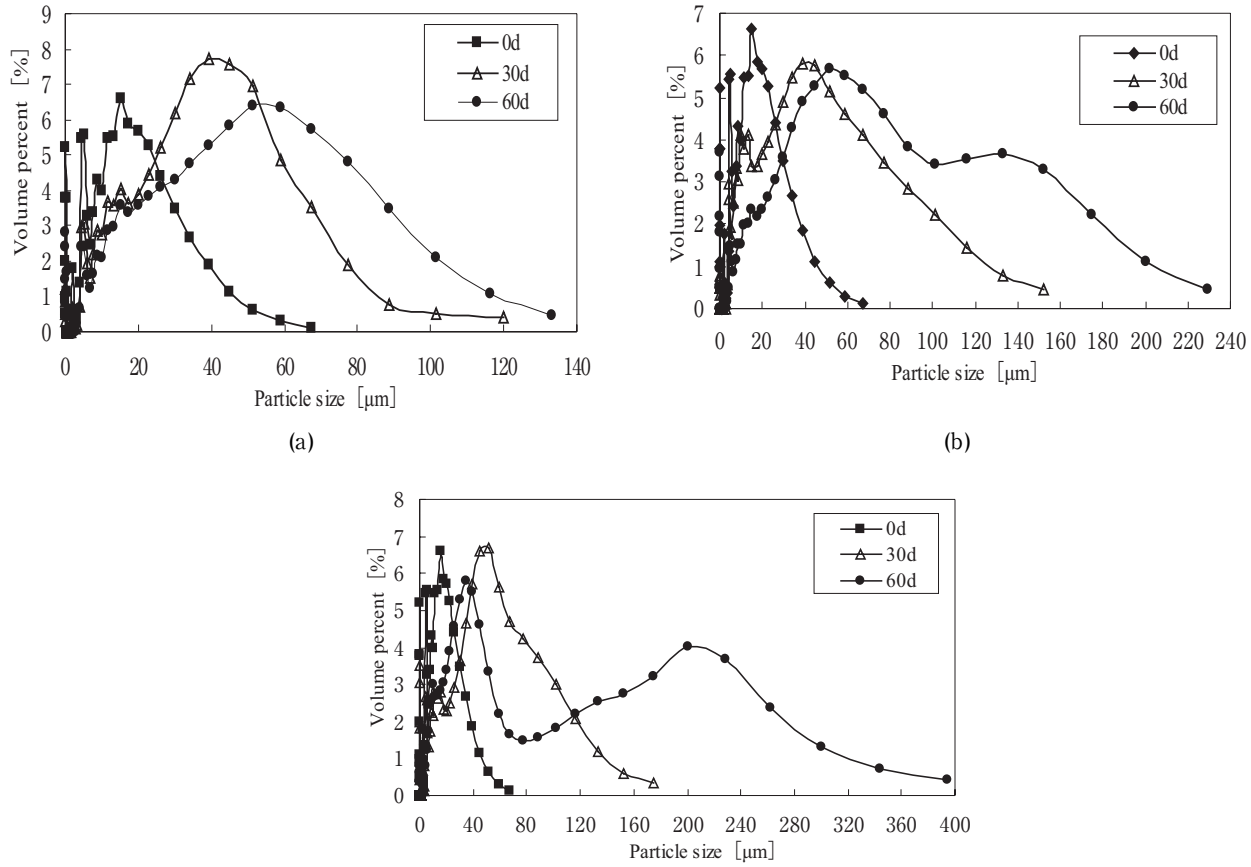
The stability of coal tar pitch powder was investigated with particle distribution and the images analysis of SEM. The coal tar pitch powder with size less than 200 mesh was preserved in different temperature and time with 1.5% AES. There were many factors, such as particle size, save time, and storage temperature, that had effects on the stability of coal tar pitch. The main factor was the changing of the distribution of particle size.

The coal tar pitch powder prepared without dispersants, whose particle size less than 200 mesh, were respectively preserved at 20°C, 30°C and 40°C for 30 or 60 days. Their stability was investigated by the particle size distribution instrument, and the results were shown in Figure 2. Figure 2 showed that the particle size distribution of the coal tar pitch powder was widened obviously after being saved at 20°C, 30°C and 40°C. The longer save time last, the wider particle size distribution would be. Some large particles appeared after being preserved for 60 days. It suggested that the stability of coal tar pitch powder became worse when temperature and time increased.

The high temperature coal tar pitch powder with size less than 200 mesh was prepared with 1.5 % AES, which was respectively preserved at 20°C, 30°C and 40°C for 30 or 60 days. And then its stability was investigated by the particle size distribution instrument. The results can be seen from the Figure 3 that comparing with newly-made coal tar pitch powder, the distribution of particle size, which was prepared with 1.5% AES, was widened as the time went by when preserved at 20°C. However, the particle size changed sharply as time went by at 30°C or 40°C, at the same time a mount of large particles appeared. It showed that the stability of coal tar pitch powder weakened with the increasing of temperature and time.

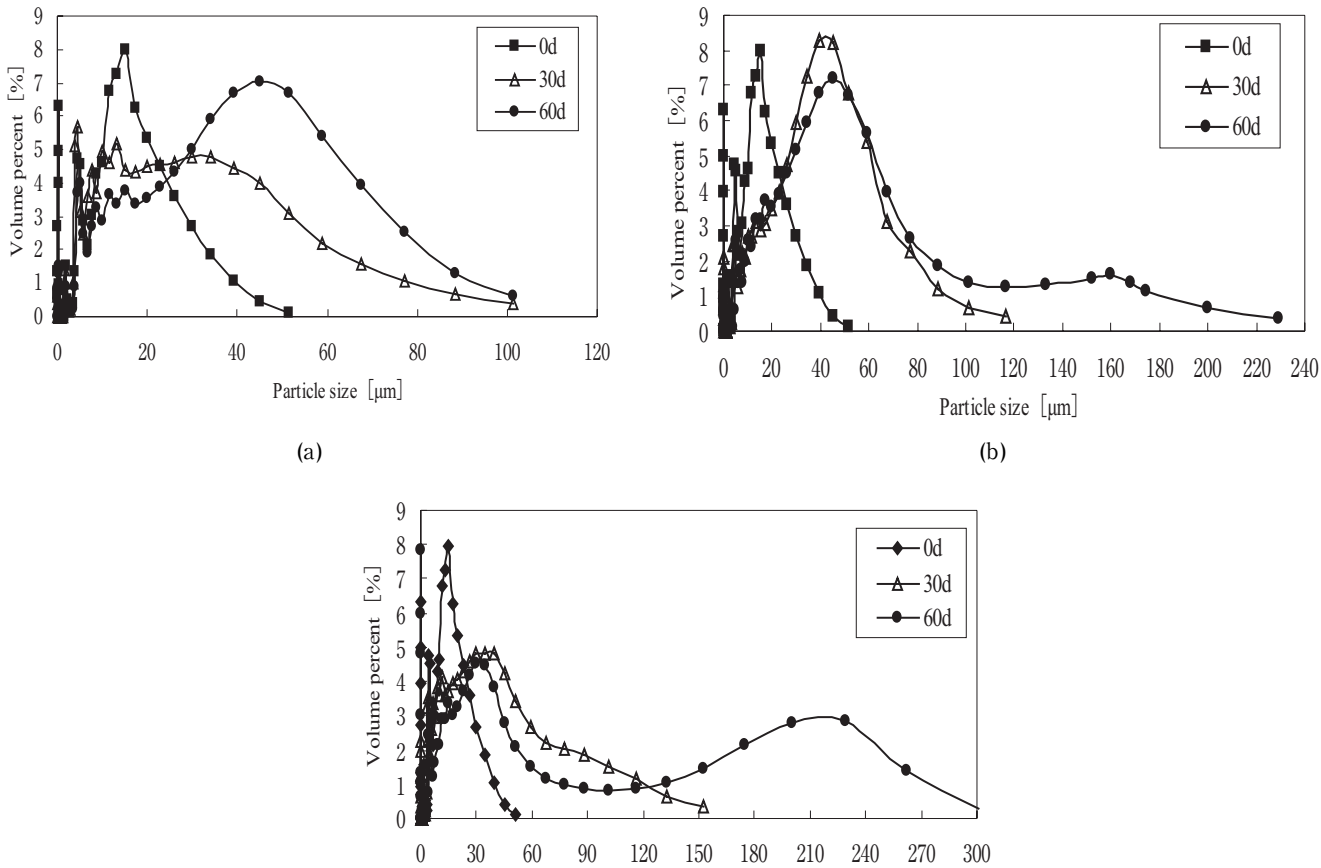
The coal tar pitch powder's particle size, which was prepared with 1.5% AES, showed a slight increasing when the temperature increased. And the particle size had greatly changed with the increasing of temperature when preserved for 60 days. At the same time a certain percentage of big particles appeared. It suggested that the stability of coal tar pitch powder weakened with the increasing of temperature.

The influence of AES on the stability of coal tar pitch powder, whose particle size was less than 200 mesh, was



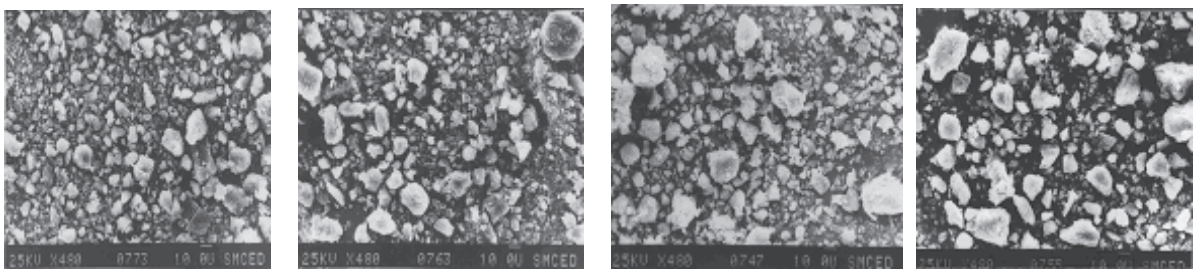
(a) 20°C ; (b) 30°C ; (c) 40°C

Figure 2 Granularity distribution of the blank samples conserved for different time.



(a) 20°C ; (b) 30°C ; (c) 40°C

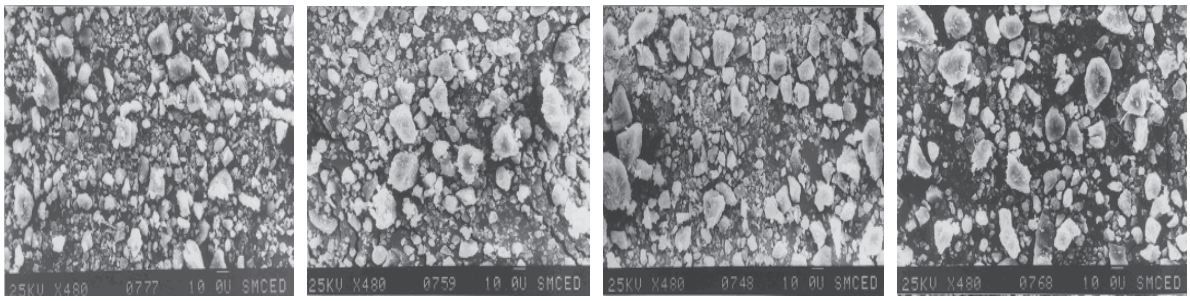
Figure 3 Granularity distribution of the samples adding 1.5%AES for different time.



(a) (b) (c) (d)

(a) The newly-prepared; (b) 20^{0}C; (c)30^{0}C; (d)40^{0}C

Figure 4 The blank sample conserved for 30 days.



(a) (b) (c) (d)

(a) The newly-prepared sampleAES; (b) 20^{0}C; (c)30^{0}C; (d)40^{0}C

Figure 5 The sample adding 1.5%AES conserved for 30 days.

studied. the particle size and distribution of sample without any dispersant and the sample with 1.5% AES addition were analyzed in different preservation time and temperature on the basis of the new sample by SEM, the results were shown in Figure 4 and Figure 5.

It can be seen from Figure 4 that there was no obvious difference in particle size and distribution between the coal tar pitch powder, whose particle size was less than 200 mesh without dispersants addition, and the new-made one after being preserved at 20°C, 30°C and 40°C for 30 days. It can be seen from Figure 5 that there was not obvious difference in particle size and distribution between them.

4. Conclusion

The present paper reports that the coal tar pitch powder prepared by cryogenic grinding technology, preserved at 20°C and 30°C for 60 days, has a relatively low particle size and more stability. Especially with 1.5% AES addition, the distribution of the particle of the coal tar pitch powder will be better. The results showed that the stability and distribution of the coal tar pitch with suitable dispersants can be improved effectively.

5. Acknowledgment

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