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Study on the Dispersion of 1.5mm & 4mm Chopped Carbon Fiber in Triethyl Phosphate

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Abstract. For the preparation of chopped CF (carbon fiber, CF) composite material, it must be scattered at first. Based on the theory of "direct observation" and "crowding factor", 1.5 mm and 4 mm short cut dispersion of CF which is done by acetone firstly in triethyl phosphate was studied in this paper, using triethyl phosphate as dispersant. Controlling the dosage of the dispersant and ultrasonic time to prepare for the dispersion of suspension, the macroscopic and microscopic structure of CF were tested before and after treatment. Test results show that to achieve good dispersibility for 4mm and 1.5mm CF with mass of 0.1g, the ultrasonic time should be controlled 2h and the amount of triethyl phosphate, the quality is 0.1 g of 4 mm and 1.5 mm of CF good dispersibility, respectively is 200 ml and 300 ml of dispersion conditions.

1 Introduction

Carbon fiber, as the representative of carbon series materials, is usually prepared into composite material for its features of low weight and price, good mechanical property etc. At present, it is widely used in the fields of building, chemistry, industry and aerospace etc. In the process of preparing composite materials, the problem of CF（carbon fiber, CF）dispersion in liquid medium has always been a difficulty. Many scholars at home and abroad have carried out extensive research on it and made great achievements[1~4]. With the target to form hydrophilic, oxygen-containing functional group on the surface of CF to improve its hydrophily[6], silane coupling agent, strong oxidant and chemical reagent, are usually used as three kinds of dispersing agent[5] for it. On the surface of CF ,then improve its hydrophily[6]; traditional evaluation methodology on CF dispersion are: the turbidity value method[4], the surface charge method[4], the resistivity test method[2] and the dispersion ratio method[1] , but the technological process is redundant and experimentation is complex. Based on such a situation, we have realized the CF dispersion by adopting triethyl phosphate as the dispersing agent, which has simple process and is easy to operate. We used methods of combing direct observation with 'crowding factor' to evaluate the dispersing performance of CF and made an analysis to its dispersing mechanism.

2 Experiment

2.1 Experiment Materials and Instruments

The carbon fiber in the experiment is produced by Yancheng Xiangsheng Carbon Fiber Products Co., Ltd., whose specific parameters are as follows: 1.5mm,4mm CF（diameter is about 7μm），the tensile strength of 3800MPa, the tensile modulus of 228GPa,the density of $1.75 g/cm^3$ and the electrical resistivity of $(1.0\sim1.6)\times10^{-3}\ \Omega\cdot cm$. We choose acetone(AR, Tianjin Guangfu Reagent Co.,500ml）to conduct pretreatment on CF. Triethyl phosphate is selected as the dispersing agent (CP, Tianjin Guangfu Reagent Co., 500ml). AE200 analytical balance (That accuracy is 0.0001g). KQ-250DB type numerical control supersonic cleaner(with temperature control range at 20~80℃，the ultrasonic electric power of 100~250W，ultrasonic cleaning time of 1~480min， Kunshan Ultrasonic Instruments Co., Ltd.).

3 Test Method

3.1 CF Pretreatment

Pretreatment is aimed at removing impurities on the fiber surface. First，we weigh appropriate 1.5mm and 4mm CF with the AE200 analytical balance; put them in the acetone solvent for soaking 2h in normal temperature; then, use ionized water to clean several times until the liquor is clear, finally we get the pretreatment products after natural drying.

3.2 The CF Dispersion in Triethyl Phosphate

Put the 1.5mm and 4mm CF that experienced pretreatment in 500ml- beakers respectively, and use the ultrasonic vibration to achieve dispersion then finally

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determine the optimal disperse conditions by adjusting the amount of CF and triethyl phosphate and controlling ultrasound time, through direct observation. Figure.1 and 2 are respectively the photo of 1.5mm and 4mm CF that before and after pretreatment.

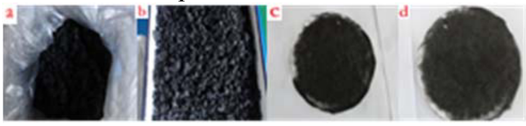

Figure 1. 1.5mmCF before and after pretreatment

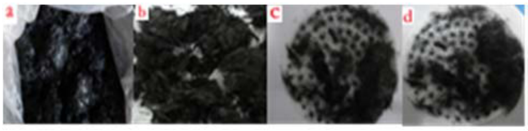

Figure 2. 4mmCF before and after pretreatment

3.3 Evaluation on Dispersion

The commonly used evaluation method is mainly dispersion ratio method[1], which is easy to calculate but complex for the experiment process to operate. In order to realize CF dispersion through simple process, we hereby shall adopt characterization method of dispersing performance for aramid fiber in references[7], using methods of combining direct observation and crowding factor calculation[8] to give a comprehensive assessment on the homodisperse state of CF's suspendability in triethyl phosphate. This method is easy to operate and hasn't been reported in CF dispersion. The method of direct observation is to observe suspension disperse condition in a visual inspection way. 'Crowding factor' is defined as the number of fiber in spherical medium (whose diameter equals the length of singer fiber) and represented by ***N***. The definition is shown in formula (1) .When $1 < N < 60$[7], the dispersion of fiber in dispersing agent is good; therefore, 'crowding factor' can be used in quantitative description to fiber's dispersion situation.

$$N = \frac{2}{3} \cdot C_v \cdot \left(\frac{L}{d}\right)^2 \quad (1)$$

Where,

N represents crowding factor, non-dimensional
C_v is the volume concentration of fiber, %;
L is the length of single CF, mm;
d is the diameter of single CF, mm.

4 Experiment Results and Analysis

4.1 The Dispersion of CF in Triethyl Phosphate

The dispersion condition for the "crowding factor" of CF in 100ml, 200 ml and 300ml was shown from Table 1.

Length	Mass	Triethyl phosphate	N
1.5mm	0.05g	100ml(fig. g)	9
		200ml(fig. h)	3
		300ml(fig. i)	2
1.5mm	0.1g	100ml(fig. j)	18
		200ml(fig. k)	9
		300ml(fig. l)	3

Length	Mass	Triethyl phosphate	N
4mm	0.05g	100ml(fig. a)	65
		200ml(fig. b)	22
		300ml(fig. c)	21
4mm	0.1g	100ml(fig. d)	131
		200ml(fig. e)	65
		300ml(fig. f)	22

Table 1. The Dispersion Condition For The 'Crowding Factor' Of 1.5 Mm& 4mm Cf

From pictures of Figure.3 (fig.***a*** to fig.***l***), we could discover that: even though under the circumstance that being with same N, the dispersion condition is inconsistent cause of different ultrasound time. According to the definition formula of crowding factor, we can get to know, in the same situation, the length of CF is the main factor that influencing dispersion for CF with equal quality. That is, the bigger for the draw ratio, the more beneficial to the CF dispersion. This is because, the collision times for single CF increased with the function of ultrasound, and the shorter for single CF, the better for its dispersity as mutual repulsion of surface charge. When the single CF achieved balance in dispersion liquid, the surface charge would gather again and appear the phenomenon of second aggregation. Finally, we determined the optimal experiment condition based on methods of direct observation and 'crowding factor 'calculation. Test results show that the ultrasonic time 2h and the amount of triethyl phosphate, respectively is 200 ml and 300 ml of dispersion conditions, the quality is 0.1 g of 4mm and 1.5mm of CF good dispersibility.

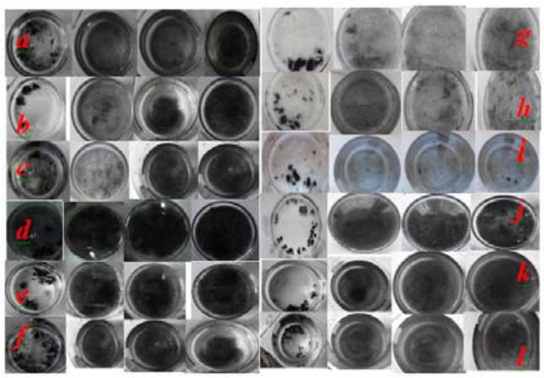

Figure 3. 1.5mm&4mm CF before and after pretreatment

4.2 The SEM Test Analysis Before and After CF Dispersion

The surface of new purchased CF takes on regular striation with obvious crack and groove. The kind of structure makes CF be difficult to spit into single ones

[6].Impurities on the surface of 1.5mm and 4mm CF were removed after pretreating by acetone, and original regular strips were destroyed with the appearance shown in Fig.4-a and Fig.4-b).After treating with triethyl phosphate on the basis, the streak and crack on the CF surface were wrapped and filled （see Fig.4-c and Fig.4-d），now, the surface of CF became bright and clean ,more hydrated than before, the dispersity of CF in the solvent was improved consequently.

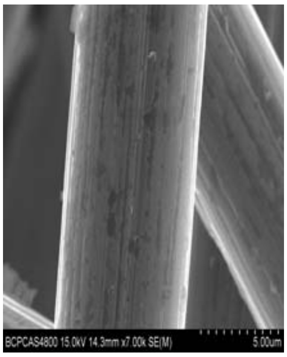

Fig. 4-a

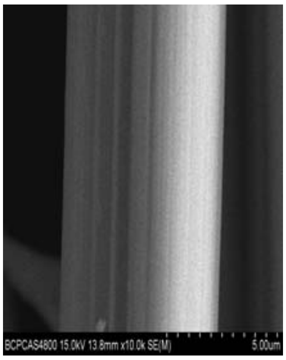

Fig. 4-b

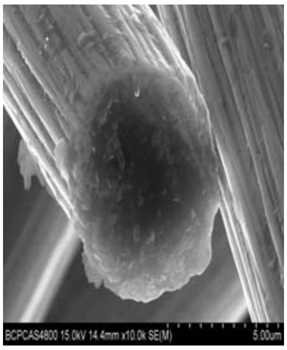

Fig. 4-c

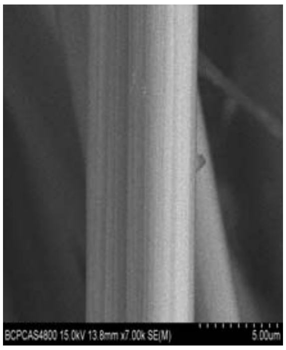

Fig. 4-d

4.3 Mechanism Analysis on How Triethyl Phosphate Improved the Dispersion of Short CF

According to the study of Heyongjia et al, PAN-based carbon fiber could form similar colloidal dispersion system by adsorbing polar bond, such as hydroxyl or carbonyl polar group, to attain the dispersion. In the triethyl phosphate solution, a lot of carboxide formed a skim on the surface of the CF, after a time of ultrasonic function, the repulsive force between each CF surface strengthened, and the dispersion of CF was consequently improved. With the increase of ultrasound time, optimal dispersion effect realized when the repulsive force of single CF surface reached balance in triethyl phosphate solution.

5 Conclusion

Experiments results show that the dispersity of 4mm and 1.5mm CF (with mass of 0.1g)is good under the disperse conditions of 2h of ultrasound time and triethyl phosphate addition are of 200 and 300ml respectively. We can theoretically determine the dispersity of short cut CF in organic solvent through “crowding factor” method. As the actual operation is influenced by ultrasound time [5], a lot of charge gathered on short CF surface will lead to second aggregation phenomenon, so it still needs combining with the observation method to finally determine the optimal situation of dispersion.

References

1. Wangchuang,Likezhi,Lihejun. Dispersity of Short Carbon Fiber in Different Dispersing Agents [J]. Fine Chemicals, 1, (2007)1-4.
2. Heyongjia，Jinshun，Lvlinnv.The Effect of HPMC on the Carbon Fiber Dispersity[J].Function Materials, 6, (2010)1034-1037.
3. Wangbaomn,Mahainan,Zhangyuan et al. The Surface Finish of Carbon Nanofiber and its Dispersion in Water Solution[J]. Materials Science and Technology, 4(2014) 41-48.
4. Cuiying, Sunyu,Wuwo et al. The Effect of Coating Treatment of Dispersing Agent on Dispersing Performance of CF in Oily Body [J]. Surface Technology,1(2015)， 112-116.
5. Lizhao. Ultrasonic Testing and Evaluation Technique Study on the Porosity of Carbon Fiber Composite [D]. Hangzhou：Zhejiang University. 2014
6. Zhaojun,Hujian, Liangyun et al. Surface Properties of Carbon Fiber and its Dispersity in Water[J]. China Papermaking,5(2008) 15-18.
7. Liujunhua. The Dispersity of Domestic Para-aramid Fiber Suspension System & its Mechanism Study [D]. Xi’an：Shaanxi University of Science and Technology.2014
8. Zhangzeng,Hujiang. Evaluation on the Disperstiy of Fiber [J]. Paper Science & Technology. 5(2001) 30-31.
9. Heyongjia，Jinshun，Lvlinnv.The Effect of HPMC on the Carbon Fiber Dispersity[J].Function Materials, 6(2010)1034-1037.