Study on the Fiber Quantitative Analysis for Polysulfonamide High Temperature Filtration Material

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Abstract: This paper focused on Polysulfonamide(PSA) high temperature resistance filtration material, the burn characteristics, appearance, dissolution characteristic and infrared spectroscopy had been studied for three commonly used fibers(PSA, PI, PTFE). The qualitative identification method was got by comprehensive judgment. And on this basis, the quantitative analysis methods had been discussed for PSA blended product with PI or PTFE or PI and PTFE. The verification test showed that the quantitative analysis method was accurate and reliable, could be used to fiber content test for PSA blended filtration material.

1 INTRODUCTION

Polysulfonamide (PSA), a kind of aromatic polyamide that macromolecular main chain contains sulfuryl (-SO2-), is usually obtained through reaction in solution condensation by 4, 4'- diamino diphenyl sulfone (DDS) and paraphthaloyl chloride (TPC). It not only has good physical and mechanical properties, but also has good heat resistance, fire resistance, electrical insulation, corrosion resistance, radiation resistance and dimensional stability. For the anti-thermal oxidation aging performance, PSA significantly exceeds the aromatic polyamide fiber, such as aramid 1313 and aramid 1414. In terms of improving environmental protection and three wastes treatment, as the 200-250℃ high temperature flue gas filtration material, it has been widely used. At the same time, it is also used as heat resistant, flame-proof fabric, high temperature resistant insulation paper.

At present, the research on PSA are mainly focused on the manufacture and industrialized application, especially in the industrial community[1-2]. Domestic inspection agencies do not have a systematic method for quantifying PSA fiber in related high temperature filtration material. With the rapid development of industry economy, the application of PSA fiber has become more and more extensive, and the identification technology of PSA fiber and other fibers is becoming more and more urgent. This paper focuses on high temperature filtration composite materials, which are usually made by Polysulfonamide (PSA), polyimide (PI) [3-4] and polytetrafluoroethylene (PTFE). In this paper, the burning behaviour, appearance, chemical solubility and infrared spectra of three kinds of fibers were discussed, and the quantitative analysis method of PSA fiber and other two fiber blended products was also studied. It provides a reference for the qualitative identification and quantitative analysis method of high temperature filtration material for PSA fiber.

2 EXPERIMENT

2.1 Reagent and Instrument

Sulfuric acid, hydrochloric acid, nitric acid, sodium hypochlorite, sodium hydroxide, formic acid, ice acetic acid, potassium thiocyanate, hydrofluoric acid, cupric hydroxide, ammonium hydroxide, N, N-dimethyl formamide, acetone, tetrahydrofuran, phenol, carbon tetrachloride, arsenic ikari, 1, 4-butyrolactone, dimethyl sulfoxide, cyclohexanone, methylene chloride, dioxane, hydrogen peroxide, xylene, etc. All the above reagents are analytical pure.

Soxhlet extractor, thermostatic water bath, analytical balance (accuracy is 0.0001g), triangle flask with a plug, and glass vessels etc.

2.2 Sample and Pretreatment

PSA is provided by Shanghai Tanlon Fiber co., ltd. P84(one of PI fiber) was purchased from evonik industries-specialty chemicals (Shanghai) co., LTD., PTFE was purchased from Zhejiang Kertice Hi-Tech Fluro-Material co,ltd. Three kinds of fibers are short fiber. The sample was placed in the soxler extractor, and was extracted for 1 hour with ethylether. After the ether volatilization in a sample, the sample was soaked in cold water for 1 hour, and then was dehydrated and dried.

3 TEST RESULTS AND DISCUSSION

3.1 Burning Test

3 fibers' burning behavior were showed in table 1.

Table 1 3 fibers' burning behaviour.

sample	burning behavior			smell	residue
	close to flame	in flame	away from flame		
PSA	no melt no Shrink	melt,shrink and brun	self-extinguishing	little paste flavour	like black coke
P84	no melt no shrink	reddish	self-extinguishing	little spicy flavor	black ash
PTFE	melt and shrink	shrink and burn	self-extinguishing	little paraffin flavour	little black power

3.2 Microscopic Examination

3 fibers' longitudinal and cross sections were showed in figure 1. As can be seen from figure 1, the longitudinal section of PSA is smooth and grooved, and cross-section is close to circle. The longitudinal section of P84 is smooth and grooved, and the cross-section is trifoliate. Most PTFE fiber's longitudinal section is like flat belt, which has obvious long stripe and horizontal stripes. The fiber diameter difference is big, which has split ends. The cross-section is close to oval and irregular polygon.

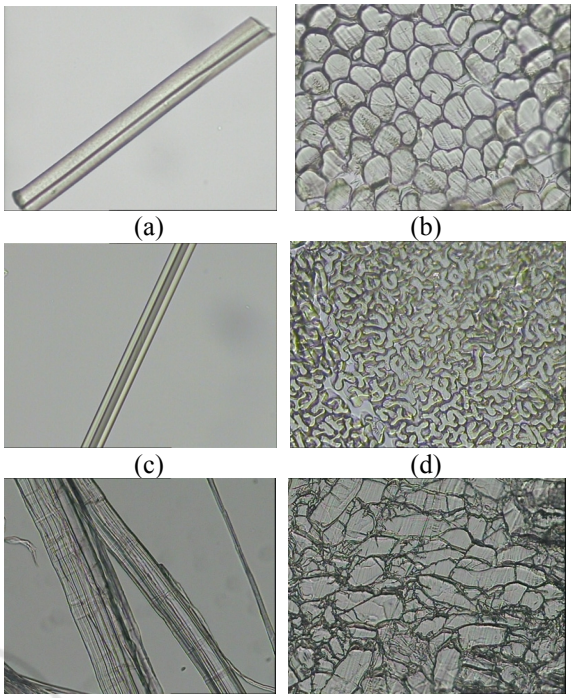

Fig.1 3 fibers' longitudinal and cross section.(a)and (b)is PSA, (c) and (d) is P84, (e) and (f) is PTFE .

3.3 Dissolution Test

3 fibers' Chemical solubility were showed in table 2.

reagent	room temperature			boiling		
	PSA	P84	PTFE	PSA	P84	PTFE
95%-98% sulfuric acid	S	S	I	S	S	I
75% sulfuric acid	I	I	I	S	I	I
36%-38% hydrochloric acid	I	I	I	I	I	I
20% hydrochloric acid	I	I	I	I	I	I
1mol/L sodium hypochlorite	I	I	I	I	I	I
5% sodium hydroxide	I	I	I	I	I	I
65%-68% nitric acid	I	I	I	S	S	I
88% methane acid	I	I	I	I	I	I
99% acetic acid	I	I	I	I	I	I
hydrofluoric acid	I	I	I	-	-	-
copper ammonia	I	I	I	I	-	-
65% potassium thiocyanate	I	I	I	I	I	I
tetrachloride	I	I	I	I	I	I
xylene	I	I	I	I	I	I
dimethylformamide (DMF)	S	I	I	S	S	I
acetone	I	I	I	I	I	I
tetrahydrofuran	I	I	I	I	I	-
phenol	I	I	I	I	I	I
phenol/ tetrachlorethane	I	I	I	I	I	I

1, 4- butylene	I	I	I	I	I	I
dimethylsulfoxide (DMSO)	S	I	I	S	I	I
cyclohexanone	I	I	I	I	I	I
dichloromethane	I	I	I	I	I	I
dioxane	I	I	I	I	I	I
ethyl acetate	I	I	I	I	I	I
pyridine	I	I	I	I	I	I
Note1：symbol description S-- soluble，I—Insoluble.						

3.4 Infrared Test

Take the right amount of samples and place them directly on the germanium crystal, rotate the fixed button of OMNIC sampler to pressure the sample, the attenuated total reflection (ATR) was used to get the attenuated full-reflection infrared spectrogram of the sample. The instrument test condition is as follows: detector is dtgs-kbr; The wave number resolution is 4 cm-1; the scanning number is 32 times; The wave number ranges are from 600 cm-1to 4000 cm-1.

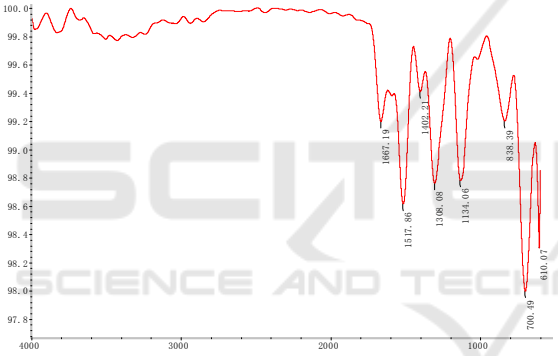

Figure 2. infrared spectrogram of PSA.

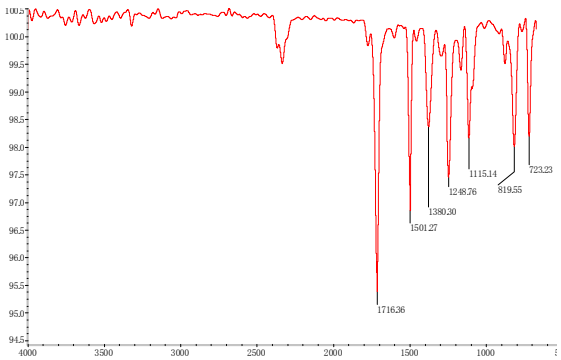

Figure 3. infrared spectrogram of P84.

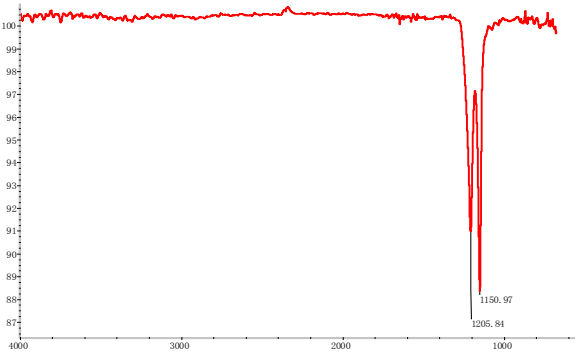

Figure 4. infrared spectrogram of PTFE.

In figure 2, the absorption peak at 1667 cm-1 is the stretching vibration of C=O; the peak at 1517 cm-1 is the benzene ring's stretching vibration. The two strong peaks near 1308 cm-1 and 1131 cm-1 belong to the asymmetric and symmetric stretching vibration peak of the sulfone group. In figure 3, the peak at 1778cm-1 and 1716 cm-1 are asymmetric and symmetric stretching vibrations of C=O from imide ring, peak at 1381cm-1 belongs to stretching vibration of C-N; peak at 724cm-1 belongs to bending vibration of C=O from imide ring.Figure 4 shows that PTFE has two strong peaks around 1210cm-1 and 1150 cm-1. The peak around 1210cm-1 is asymmetric stretching vibration of CF2, the other is symmetric stretching vibration of CF2. The absorption peak of CF2 group is the strongest in the spectrum of PTFE, which can also prove that CF2 is the basic unit in the molecular chain.

3.5 Qualitative Analysis

In this paper four kinds of method burning test, microscopic examination, chemical dissolution, infrared spectrum test were used to identify above three kinds of fibers. Burning test and microscopic examination can only preliminary judge the fiber category, and can't tell what it is. Combined with chemical dissolution test, experimenter basically can determine whether it contains in textiles. With the increasing variety of fibers, Fibers with similar dissolving properties may also constantly spring up. Chemical dissolving method has its limitation. Infrared spectroscopy was useful to further confirm what it is. Therefore, from a variety of possibilities, the above four methods were used to study the qualitative analysis methods comprehensively. Experimenters can fully identify which fiber it is and quantify fiber content.

3.6 Quantitative Analysis

3.6.1 Two Component Fabric of PSA and P84

Method using dimethylformamide(DMF).Dissolving the PSA from known weight drying sample by dimethylformamide, collecting residue, cleaning, drying and weighing. The PSA mass content in sample was calculated. The other fiber is P84, which mass content could also be got.

Method using dimethylsulfoxide(DMSO). Dissolving the PSA from known weight drying sample by dimethylsulfoxide, collecting residue, cleaning, drying and weighing. The PSA mass content in sample was calculated. The other fiber is P84, which mass content could also be got.

3.6.2 Two Component Fabric of PSA and PTFE

Method using DMF and DMSO could also be used. Dissolving the PSA from known weight drying sample, collecting residue, cleaning, drying and weighing. The PSA mass content in sample was calculated. The other fiber is PTFE, which mass content could also be got.

Method using 95-98% sulfuric acid Dissolving the PSA from known weight drying sample by 95-98% sulfuric acid, collecting residue, cleaning, drying and weighing. The PSA mass content in sample was calculated. The other fiber is PTFE, which mass content could also be got.

3.6.3 Tri-component Fabric of PSA, P84 and PTFE

When faced with tri-component fabric or needle punched nonwovens of PSA, P84, we have two steps. First, dissolving the PSA from known weight drying sample by DMF, collecting residue, cleaning, drying and weighing. Second, dissolving the P84 from known weight drying residue by boiling DMF, recollecting residue, recleaning, redrying and reweighing.The PSA mass content in sample was calculated. The other fiber is PTFE, which mass content could also be got.

Using above methods (3.6.1-3.6.3), the some samples that known mass fraction were tested by experimenter A and B. The parallel test results were showed in table 3.

sample	reagent	Mass content	Parallel test results		Max error
			experimenter A	experimenter B	
PSA/P84	DMF	60.45/39.55	60.87/39.13	60.21/39.79	60.75/39.25	60.34/39.66	0.32
	DMSO	60.45/39.55	60.19/39.81	60.39/39.61	60.85/39.15	60.57/39.43	0.40
PSA/PTFE	DMF	52.88/47.12	52.97/47.03	51.51/48.49	52.99/47.01	52.78/47.22	0.37
	DMSO	52.88/47.12	52.65/47.35	52.99/47.01	52.53/47.47	52.71/47.29	0.35
	95-98% H2SO4	52.88/47.12	52.73/47.27	52.70/47.30	52.93/47.07	52.78/47.22	0.18
PSA/P84/PTFE	DMF	30.25/35.42/34.33	30.25/35.42/34.33	30.25/35.42/34.33	30.25/35.42/34.33	30.25/35.42/34.33	0.89

From table 3, in different experimental conditions, the stability in chemical reagents of polysulfonamide fiber and other fibers blended product is good. The test result error of experimenter A and B is no more than 1%, which is conform to meet the requirements of GB/T 2910-2009 testing standard. While using solvent to dissolve objective blended products, the stability of the parallel test performance is good, that was according with the requirements of quantitative analysis. The quantitative test results in table 3 showed that the dissolving methods of polysulfonamide fiber and other fibers blended products are feasible and valid.

4 CONCLUSIONS

In this paper, the four kinds of testing methods, such as the burning test, microscopic observation, chemical dissolution, infrared spectrum and so on, were used to analyze 3 kinds of fiber (PSA, PI and PTFE) comprehensively, which were commonly used in high temperature filter material. A comprehensive study on the qualitative analysis methods of three kinds of fiber was carried out that so as to realize the qualitative identification for three fibers. On this basis, according to the chemical dissolution characteristics, establish the quantitative analysis methods were established. Through the known mass content blended samples, The test result error of experimenter A and B is no more than 1%, which is conform to meet the requirements of testing standard. The stability of the parallel test performance is good. The dissolving methods of PSA, PI and PTFE blended products are feasible and valid. The testing results are accurate and reliable.

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