Yongjian XU Leigang ZUO Xue Li Faping CHAI

1.College of Light Industry and Energy, Shaanxi University of Science and Technology;
2.Shaanxi Province of Key Lab of Paper Technology and Specialty Paper, Xi'an, 710021 China

Preparation and characterization of Glass Fiber Reinforced Sound Absorption and Flame Retardant Paper

Abstract

The preparation process of glass fiber reinforced sound absorption and flame retardant material was investigated by measuring the basis weight, tensile strength and flame retardant grade indexes. The effects of binder and flame retardant type and loading on the paper properties were explored. SEM was employed to characterize the distribution and reaction mechanism of binder and flame retardant on the paper. The results showed that binder B was more effective than binder A. At 4% concentration of binder B for the basis weight of paper 44.55 g/m^2, the tensile strength achieved up to 15.52 N/15 mm. when the concentration of flame retardants C was 5%, the effect of flame retardant C was found to be the best: at this concentration, the basis weight of paper was 43.98 g/m^2, and the flame retardant grade could reach to level 1. Studies were performed on the ratio of the binder and the flame retardant to find the effects on flame retardant. Accordingly, when the ratio of binder C to flame retardant was 3:7, the paper basis weight , tensile strength and flame retardant grade was 60.00 g/m^2, 15.10 N/15 mm, level 1, respectively. While the ratio of blinder C to flame retardant was 4:7, the material basis weight was 60.00 g/m^2, tensile strength was 15.10 N/15 mm and flame retardant grade reached level 1. Under these ratios, all indexes reached the standards of paper performance.

Key words: *Glass fiber, Binder, Flame retardant, Sound absorption and flame retardant material*

Introduction

Glass fiber is one of the main materials in acoustic engineering. It has beneficial effects on sound adsorption, noise reduction and improving the quality of indoor sound. The sound adsorption coefficient is a parameter to judge the sound adsorption performance of the material. Under a certain frequency, sound adsorption coefficient is associated with transmission of sound energy flux, transmission of absorbed radiation energy flux and transmission of incident sound energy flux [1]. Noise reduction coefficient is an important parameter for evaluating the quality of sound absorption materials. It is usually defined the arithmetical mean of adsorption coefficient in 250Hz, 500 Hz, 1000 Hz and 2000 Hz. Noise reduction coefficient of glass fiber sound absorption board is generally greater than 0.40, and even can reach more than 0.80 [2]. The sound adsorption performance is intrinsic characteristic of glass fiber, so the original paper is made of the glass fibers and plant fibers, then adding adhesive and flame retardant to obtain the glass fiber reinforced sound absorption and flame retardant paper which has the properties with fire prevention and sound adsorption. The paper which is dyed by black slurry has decorative performance. It can attach at the back of aluminum ceiling, mineral wool and glass surface. It is wildly used in cinema, home, open air traffic equipment and air condition.

To develop different functional materials containing glass fibers using wet papermaking technology could not only meet the needs of industrial production and daily life, but could also solve the problems of the shortage, high price and poor durability of plant fibers [3].Meanwhile, it also enriched the varieties of production, improved the properties and speeded up the development of technology and economy. Glass fiber was one of main materials used in acoustic engineering. It has a good performance on sound absorption, noise reduction, sound insulation and improving acoustic fidelity of room [4]. Noise reduction coefficient was an important parameter for evaluating the quality of sound absorption materials [5]. Noise reduction coefficient of glass fiber acoustic board was generally between 0.40 and 0.80. Owing that the sound absorption property of glass fiber was an inherent characteristic of the material [6-8]. In this study, the base paper was made by glass fibers and plant fibers, and enhanced tensile strength was obtained by coating binder. Then the flame retardant property was introduced into paper through the addition of flame retardant. Finally, the glass fiber reinforced paper with characteristics of sound absorption and flame retardant was obtained.

Experimental

Raw materials

Glass fiber was kindly supplied by Tai Shan Glass fiber Co. LTD, Shandong, China. Length: 6 mm. Diameter: 9μm.

Binder A(Solid content: 45%, Styrene-butadiene latex) was kindly supplied by Xi'an Dao Er Da Chemical Co. LTD, Shaanxi, China.

Binder B(Solid content: 45%, Expoxy resin) was kindly supplied by Xi'an Dao Er Da Chemical Co. LTD, Shaanxi, China.

Flame retardant A (Solid content: 30%, Halogen-free flame retardant) was kindly supplied by Xi'an Dao Er Da Chemical Co. LTD, Shaanxi, China.

Flame retardant B (Solid content: 98%, Halogen-free flame retardant) was kindly supplied by Shanghai Xiangmeng Chemical Co. LTD, Shanghai, China.

Flame retardant C (granular fabric flame retardant-2124) was kindly supplied by Yantai Eiffel Chemical Co. LTD, Shandong, China.

Color paste (fresh beautiful black M-100) was kindly supplied by Shanghai Caiming Chemical Co. LTD, Shang hai, China.

Experimental Methods

Experimental equipments

ZQJ1 - B Sheet shaper; BJ - 1 pulping standard fluffer; C - HJY03 thickness and tightness gauge;

Z - KZ300 pendulum tensile strength tester; Type Quanta200 scanning electron microscopy (SEM); Electronic balance.

Preparation of base paper

A small amount of dispersant was added in the glass fiber. The mixture was dispersed in pulping standard fluffier at 1000 rpm for 5min.. Then according to the dry weight ratio of 30%:70% between glass fibers with coniferous wood fiber, the coniferous wood fiber was added and dispersed with 1000 rpm again. The fibers after dispersion was dehydrated and formed on the sheet shaper. Wet paper of 40 $g/m^2$was transferred to the copper net and dried at 105℃ in the oven to obtain the base paper.

Effects of binder types and loading on paper properties

The binder A and binder B were both prepared into 100mL of solutions with the concentration of 3%, 5% and 7%, respectively. Put the base paper into Buchner funnel and the solution of 3% binder A was sprayed on the base paper. Then the vacuum suction with instantaneous high strength was used to guarantee the dry degree of paper varied in the range from 20% to 30%. Base paper was put into the oven and dried at 130 ℃ . The remaining binder solutions were sprayed on the base papers and followed the steps above. The optimal binder was chosen by measuring the papers' weight and tensile strength.

Effects of types and loading of flame retardant on paper properties

The flame retardant A, flame retardant B and flame retardant C were prepared into 100mL of solutions with the concentrations of 3%, 5% and 7%, respectively. After placing the base papers on the Buchner funnel, different binder solutions were sprayed on the base papers in turn. The base paper with binder solution was pumped under instantaneous high strength of vacuum. When the dryness degree of paper reached from 20% to 30%, the base paper was put into the oven and dried at 130°. The optimal flame retardant was chosen by testing the papers' weight and tensile strength.

Effects of binder ratios to flame retardant on the paper properties

The binder B and flame retardant C was prepared into 100mL solution according to different ratios which were 4:5, 4:6, 4:7, 4:8, 3:4, 3:5, 3:6 and 3:7. The base paper on the Buchner funnel and the binder B was sprayed on the filter paper pumped vacuum filtration, firstly. When the surface of base paper was under dried without flowing binder and the dryness of paper reached to approximately 20%, the flame retardant at one of above ratios was sprayed on the base paper next. This glue method was called “two stage vacuum suction method”. The paper with binder and flame retardant was put into oven at 130°C. After drying, the papers were tested for weight, thickness, tensile strength and flame retardant property.

Analysis of binder and flame retardant effects

In the preparation process, the strength of the paper was provided by the addition of binder, while the flame retardant performance was achieved by addition of flame retardant. During measurement process, it was found that jamming effects existed between binders and flame retardant. In order to analyze the types of jamming effects, scanning electron microscope (SEM) was used to observe the distribution of binder and its performance in base paper. The possible mechanism was deduced accordingly.

Performance characterization

The quantitative method for the base paper: according to the national standard of China GB/T 451.2-2002.

Test of thickness and density: according to the national standard of China GB/T 451.3-1989.

Test of tensile strength: according to the national standard of China GB/T 451.3-1989.
Test of flame retardant performance: according to “manufacture of functional paper base paper products” China Light Industry Press, 1989.

Results and Discussion

Effects of binder types and loading on paper properties

The tensile strength of paper was influenced by the type of binder used. Generally, binder is needed to have the performances of high film strength, adhesion, water resistance and mobility. Additionally, a large loading level, mechanical and chemical stability were necessary. Different binders, were found to have different effects on paper tensile strength.

Table 1 Effects of binder types on the paper tensile strength

Binder type	concentration/ %	Weight of base paper/ g/m^2	Weight of paper $/g/m^2$	Thickness /mm	tightness/ g/m^3	Content of binder/%	Tensile strength/N/ 15mm
Binder A	3	40.42	45.69	0.194	0.235	11.53	16.84
	5	40.23	50.49	0.206	0.245	20.32	16.52
	7	40.14	54.47	0.211	0.258	25.39	19.73
Binder B	3	40.33	42.60	0.192	0.222	5.33	13.85
	5	40.57	46.18	0.202	0.229	12.15	16.70
	7	40.26	52.36	0.208	0.252	23.11	23.22

Table-1 shows the effect of two different kinds of binders (A&B) on the paper tensile strength properties. At a low concentration of binder, the effect of binder A was found to be better than that of B. With the increasing concentration of binders, the binder B was better than A. When the concentration of binder A reached to 3% , the paper tensile strength was 16.84N/15mm, while binder B was only 13.85N/15mm. However, when the concentration of binder A increased to 7%, the paper tensile strength was 19.73N/15mm, whereas the binder B of it could reach 23.22N/15mm. This was verified from the content of binder in the paper. The efficiency of binder B was higher. For instance, when the concentration of binder was 3%, the content of binder A occupied by 11.53% in the paper, the content of binder B was only 5.33%. The content of binder A was more than twice as much as binder B. While the tensile strength of paper using binder A was only 3N/15mm higher than the paper using binder B. When the concentration increased to 5% and 7%, the content of binder B was less than A in the paper, while the tensile strength of paper using binder B was stronger than the paper using binder A. As a result, the effect of binder B was better than binder A.

Table 2 indicates the effect of different concentrations of binder B on tensile strength. With the increase of the concentration of binder B, the weight of paper, content of binder and tensile strength increased. When the concentration of binder B reached to 4%, tensile strength of paper was higher than 15N/15mm, but the weight of paper was only 44.5g/m^2 and the content of binder B was 9.2% in the paper. While the concentration of binder B increased to7%, the weight of paper was only 52.36g/m^2, yet the tensile strength could reach to 23.17N/15mm.

Table 2 Effects of binder B concentrations on the paper tensile strength

concentration/ %	Weight of base paper/ g/m^2	Weight of paper $/g/m^2$	Thickness /mm	tightness/ g/m^3	Content of binder/%	Tensile strength/N/ 15mm
3	40.33	42.60	0.192	0.221	5.33	13.84
4	40.45	44.55	0.203	0.219	9.20	15.52
5	40.36	46.18	0.202	0.228	12.60	16.69
6	40.25	46.91	0.206	0.227	14.20	20.34
7	40.64	52.36	0.208	0.251	22.38	23.17

Effects of flame retardant types and its loading on paper properties

Commonly used flame retardant types including Bromic series flame retardant, Halogen - phosphorus synergistic flame retardant, Phosphorus series flame retardant and other inorganic flame retardants [9-10]. Among them, Bromic series flame retardants are the traditional flame retardant, and there are various types of Bromic series flame retardant available in the market. Low content of Bromine in the halogen-phosphorus synergistic flame retardant, thus, it is wildly used in the construction field for its advantage that the smoke is less during combustion. The application of Phosphorous flame retardant is limited for its characteristics of liquid, more volatile and poor compatibility. The halogen- phosphorus synergistic flame retardant make up for the disadvantages of phosphate ester flame retardant which are easy mobility, high volatile, thermo labile and easily aging. Inorganic flame retardants are attracted much attention due to its environmental protection and high efficiency [11]. At present, the most wildly used inorganic flame retardants are aluminum hydroxide and magnesium hydroxide flame retardants [12]. In this study, three types flame retardant, liquid flame retardant A, high viscosity liquid flame retardant B and powder flame retardant C, were chosen to study their efficiency.

Table 3 shows that different kinds of flame retardant A, B and C were added in the base paper under the same concentration, respectively. It was indicated that the flame retardant C had the best effect, while the flame retardant B was the worst. The paper was combustible up to 5% concentration of type A and B flame retardant. However, the type C flame retardant could achieve level 1 at the concentration of 5%. Secondly, when the concentration of flame retardant A reached to 7%, the flame retardant rating could reach level 2, while by using flame retardant B paper

Table 3 Effects of flame retardant types and loading on paper properties

Flame retardant type	concentration/ %	Weight of base paper/ g/m^2	Weight of paper $/g/m^2$	Thickness /mm	tightness/ g/m^3	Content of flame retardant/%	Flame retardant rating
Flame retardant A	5	39.96	42.68	0.179	0.238	6.37	combustible
	7	40.72	46.10	0.180	0.256	11.67	level 2
	9	40.61	48.94	0.182	0.268	17.04	level 2
Flame retardant B	5	40.26	43.33	0.178	0.243	7.09	combustible
	7	40.12	45.93	0.177	0.259	12.65	combustible
	9	40.18	47.15	0.174	0.271	14.78	level 2
Flame retardant C	5	39.96	43.98	0.188	0.234	9.14	level 1
	7	40.24	46.18	0.200	0.231	12.86	level 1
	9	40.07	48.70	0.215	0.226	17.72	level 1

Table 4 Effects of different concentrations of flame retardant C on flame retardant properties of paper

concentration/ %	Weight of base paper/ g/m^2	Weight of paper $/g/m^2$	Thickness /mm	tightness/ g/m^3	Content of flame retardant/%	Flame retardant rating
2	40.26	41.14	0.181	0.227	2.14	combustible
3	40.58	43.41	0.185	0.234	6.52	combustible
4	40.11	43.82	0.189	0.232	8.47	level 2
5	39.96	43.98	0.188	0.234	9.14	level 1
6	40.04	45.85	0.193	0.237	12.67	level 1

was combustible. Lastly, when the concentration of flame retardant reached 9%, by using flame retardant A and B papers could reach level 2. When the content of flame retardant C reached 9.14%, the flame retardant rating of paper was level 1. Comparing to flame retardant A and B, when they reached the maximum content of flame retardant, the flame retardant rating only reached level 2. Meanwhile, it was also found from experimental data that under the same flame retardant concentrations, the flame retardant C made the greatest contribution to the weight of paper which had indicated that the use of flame retardant C in the paper was the best. In order to find the optimum loading of flame retardant C, flame retardant C was prepared into different concentrations of solution and added into the base paper.

Table shows that the increasing concentration of flame retardant, the flame retardant rating increased from combustible rapidly up to 3% concentration to level 2 at 4% concentration and level 1 about 5% concentration of flame retardant C. When the concentration of flame retardant reached to 2% at the weight of paper was 41.14g/m^2, the content of flame retardant was 2.14% and the flame retardant rating was combustible. When the concentration reached to 4%, the weight of paper was 43.82 g/m^2, the content of flame retardant was 8.47% and the flame retardant rating of paper reached level 2. When the concentration reached to 5%, the weight of paper was 43.98 g/m^2, content of flame retardant was 9.14% and the flame retardant rating of paper could reach level 1. As a result, only did use flame retardant C, since the concentration of flame retardant reached to 5%, the content of flame retardant C could beyond 9% and flame retardant rating achieve to level 1.

Effects of binder to flame retardant ratios on the paper properties

In order to study the change of paper properties when binder and flame retardant were added at the same time. Table 5 and Table 6 presented the effects of different ratios of binder on flame retardant effect of the paper properties. In Table 5 effect of fixed concentration of binder (4%) and variation at concentration of flame retardant is presented. While table 6 effect of fixed the concentration of flame retardant which was (7%) and variation at concentration of binder is presented.

When the concentration of binder was fixed, with the increase of flame retardant concentration, the weight paper increased and the flame retardant rating

Table 5 Effects of flame retardant concentration ratios on paper properties

Binder Flame retardant	Weight of base paper/ g/m^2	Weight of paper /g/m^2	Thickness /mm	Tightness/ g/m^3	Tensile strength /N/15m	Flame retardant rating
4:5	40.36	55.28	0.218	0.253	15.24	level 2
4:6	40.24	57.56	0.226	0.255	15.76	level 2
4:7	40.57	60.00	0.236	0.254	15.25	level 1
4:8	40.44	61.87	0.245	0.253	14.25	level 1

Table 6 Effect of change of binder concentration on paper properties

Binder Flame retardant	Weight of base paper/ g/m^2	Weight of paper /g/m^2	Thickness /mm	Tightness/ g/m^3	Tensile strength /N/15m	Flame retardant rating
3:7	40.66	60.00	0.225	0.267	15.10	level 1
4:7	40.57	60.00	0.236	0.254	15.25	level 1
5:7	40.16	61.95	0.238	0.260	16.51	level 2
6:7	39.91	61.82	0.233	0.265	15.94	level 2

increased from level 1 to level 2 (Table 5). Meanwhile, the tensile strength kept at about 15N/15m and the change wasn't obvious, it was possibly because the base paper was dried directly after adding binder, the curing process of binder impacted certain strength properties in paper. Although the paper was put into water after drying, the water had little interference on the tensile strength. So the tensile strength of paper could be maintained in a better range. When the ratio of binder to flame retardant was 4:7, at the weight of paper 60g/m^2, the tensile strength of paper was found to be 15.25N/15mm and flame retardant rating was level 1.

It is shown in table 6 that when the concentration of flame retardant was 7%, with the increase of binder concentration, the weight of paper and tensile strength had changed marginally. With increase in the binder concentration from 4% to 5%, the flame retardant rating decreased from level 1 to level 2. Table 5 and Table 6 are show that change of flame retardant concentration had an obvious effect on the weight of paper. Different binder concentrations had less effect on the weight of paper, but it had an obvious effect on flame retardant rating. When the dosage of binder was increased, it had to increase the dosage of flame retardant to reach the flame

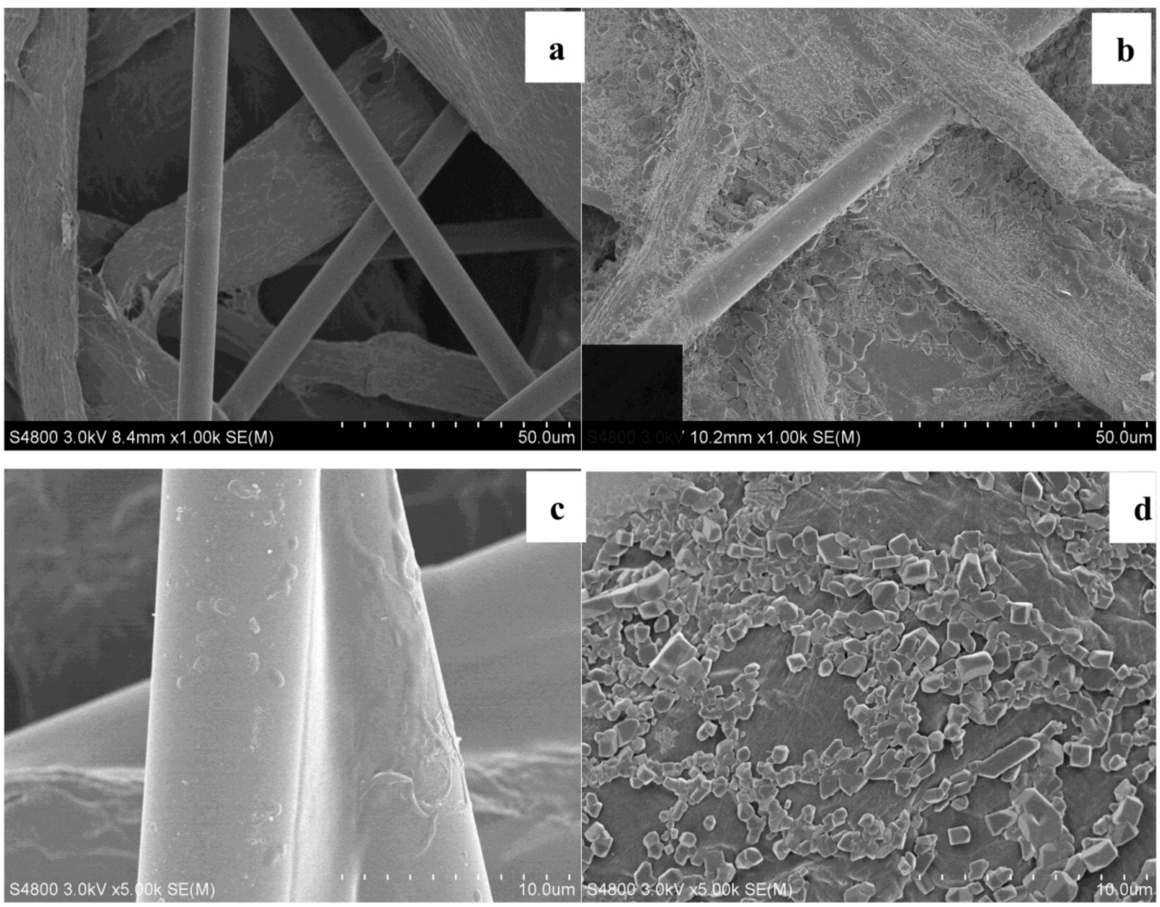

Figure 1 SEM analysis of paper

(a) Paper without binder and flame retardant
(b) Paper after dyeing
(c) Paper only with binder
(d) Paper only with flame retardant

retardant rating. As a consequence, when the ratio of binder to flame retardant was 3:7, the weight of paper was 60.00g/m^2, thickness was 0.225mm, tensile strength was 15.10N/15mm and flame retardant rating was level 1. When ratio of binder to flame retardant was 4:7, the weight of paper was 60.00g/m^2, thickness was 0.236mm, tensile strength was 15.25N/15mm and flame retardant rating was level 1. Both of these ratios could reach the standard of paper sample.

SEM analysis of paper

In general, the tensile strength of paper is generated by the binding between fibers [13-14]. SEM studies were concluded to evaluate the effect of binder and flame retardant with the plant fibers which could play an important role on flame retarding through the combination of the hydrogen bonding between single fiber. There were 70% softwood fibers in the paper, but there was not squeezing process in the preparation, meanwhile, the way of drying hindered the hydrogen bonding ability between softwood fibers. So the tensile strength was low without binder which was far from the required standard of strength. Plant fiber was able to burn; glass fiber was difficult to burn. However, binder in the paper was inflammable material, so flame retardant was needed to distribute uniformly in the fiber surface which Figure 1 shows the distribution of binder, flame retardant and color paste on the paper surface.

Figure 1(a) shows the cross distribution of glass fibers and plant fibers in the paper. Figure 1(c) shows binder which is in the form of emulsion has been absorbed on the surface of glass fibers and plant fibers, it had played an important role in filling and coherence. In figure 1(d), it is seen that flame retardant which is distributed on the surface of plant fibers and there are few parts on the glass fibers. Figure 1(d) is shown that binder filling between fibers after dyeing. It could be seen that flame retardant is absorbed on the glass fibers and plant fibers after dyeing. It might be the coating of color paste on the flame retardant which made the flame retardant to be adsorbed on the glass fibers easily. It has indicated that dyeing made more flame retardant adsorption on the glass fibers. The studies show that binder on account of adhesion enhanced the binding strength between fibers and resulted in improving the tensile strength of paper. Flame retardant being solid particles, when burnt at high temperature, hindered the combustion by released gas which hindered the burning of combustible materials.

Conclusion

1. The using effect of binder B was better than A. When the concentration reached 4%, the weight of paper was 44.55 g/m^2, tensile strength was 15.52N/15mm and the content of binder was 9.20% in the paper.

2. The using effect of flame retardant C was better than A and B. Only did use flame retardant C, when the flame retardant reached 5%, the content of flame retardant C was beyond 9%, and the flame retardant rating reached level 1.

3. Using the “two stage vacuum suction method”, the change of flame retardant concentration had an obvious effect on the weight of paper. Although the binder had no effect, it influenced the flame retardant performance. When the dosage of binder was increased, the paper had to add flame retardant to reach required standard. When the ratio of binder to flame retardant was 3:7, the weight of paper was 60.00g/m^2, thickness was 0.225mm, tensile strength was 15.10N/15mm and flame retardant rating was level 1. When ratio of binder to flame retardant was 4:7, the weight of paper was 60.00g/m^2, thickness was 0.236mm, tensile strength was 15.25N/15mm and flame retardant rating was level 1. Both of these ratios reached the standard of paper sample.

4. Glass fibers and plant fibers were in cross distribution. Binder that was in the form of emulsion was absorbed on the surface of glass fibers and plant fibers. Binder enhanced the force between fibers and improved the tensile strength of paper and played important roles on filling and absorbing. Flame retardant was solid particles. There were small parts of them absorbed on the surface of fibers.

Acknowledgment

The key laboratory project of Shaanxi science and technology department (2011HBSZS017); graduate innovation fund of Shaanxi University of Science and Technology (2014018); The National College Students' innovation and entrepreneurship training program of China（201310708011）; and Shaanxi University of Science and Technology academic leader training program (2013XSD25). They also thank Key Laboratory of Auxiliary Chemistry and Technology in Light Industry for supporting their research at Shaanxi University of Science and Technology (SUST), China.

References

[1] Jiang Zhao-zhong. Glass fiber technology. Sinopec Press, Beijing. 106-109 (2004).

[2] Liang Yu-hua, Ma Hong-cai. Glass fiber and Its Composites. Shandong Textile Science & Technology; 42 (6):47-49 (2001).

[3] Liu Ren-qing. Cellophane and glass fiber paper. Tianjin Paper Making; 32(3): 45-48 (2010).

[4] Ohsawa T, Nakayama A, Miwa M, et al. Temperature dependence of critical fiber length for glass fiber - reinforced thermosetting resins. Journal of Applied Polymer Science; 22(11): 3203-3212 (1978).

[5] Salehi-Mobarakeh H, Brisson J, Ait-kadi A. Ionic interphase of glass fiber/polyamide6,6 composites. Polym Composites; 19(3): 264-274 (1998).

[6] Aseere Lester Mickel, Leverson Amy Jean, Obernyer Kristin Franz. “Vinyl Floor Covering System with Woven or Non-woven Glass Fiber Mat Segmented Reinforcement” European Patent, EP. 1778483B1 (Set. 11 2011).

[7] Chengdong Li, Zhaofeng Chen, Jieming Zhou, et al. Material Parameter Determination in Glass Wool Mat for Sound Absorption. Applied Mechanics and Materials; 148: 1271-1275 (2011).

[8] Alan Michael Jaffee, Bowling Green. “Non-woven Glass Fiber Mat Faced Gypsum Board and Process of Manufacture” USA Patent, USP. 7842629B2 (Nov. 30 2010)

[9] Souvik Nandi. Glass-fiber Containing Composite Materials for Alkali Metal-based Batteries and Methods of Making: USA Patent, USP. 0171538A1 (Jul. 14 2011).

[10] Seiji Yoshimura. “Lithium Secondary Battery Including A Lithium Electrolyte and Glass Fiber Separator” USA Patent, USP. 7510806 B2 (Mar. 31 2009)

[11] Jaffee Alan Michael, Kajander Richare Emil. “Fiber glass mat, method and laminate” European Patent, EP. 2251474A1 (Nov. 17 2010)

[12] Pechyen C, Atong D, Aht-ong D. Effect of additives on mechanical properties and photo-stability of filament-wound glass fiber reinforced epoxy pipes. Polymers &Polymer Composites; 19(23):161169 (2011).

[13] Zhou W Y, Qi S H, Ai T, et al. Toughened epoxy resin matrix for a membrane shell by wet filament winding. Journal of Applied Polymer Science; 111: 255263 (2009).

[14] Shivamurthy B, Siddaramaiah, Prabhuswamy M S. Design, fabrication, and testing of epoxy/glass-reinforced pressure vessel for high-pressure gas storage. Journal of Reinforced Plastics and Composites; 29(15):2379-2386 (2010).