Hydrolysis of Semi-finished Polyacrylonitrile (PAN) Fiber

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Abstract

Based on above best preparation condition for semi-finished polyacrylonitrile (PAN) fiber via dry spinning at alkaline conditions, for example, hydrolyzing temperature keeps at 95℃, the mass fraction of NaOH is 15%, water treatment is 60 min, the effect of bath ratio and the recycling of the bath on fiber water absorptivity were further investigated. Furthermore, the max recovery rate of the alkali solution was discussed. The results show that bath ratio has little effect on the water absorptivity of superabsorbent fiber obtained by hydrolyzing PAN precursor. The bath can be recycled at most three times, and maxmium recovery rate of the alkali solution is about 70%.

Keywords: Polyacrylonitrile fiber, Hydrolysis, Bath Ratio, Super-absorbent fiber

1. Introduction

Super-absorbent resin shortcomings are made up because of the emergence of high absorbent fibers. Fibrous absorbent material has many advantages: (1) The use of general fiber processing machinery can be mixed with other fibers..(2) Capillarity of the increased permeability in the same time, a large surface area and fast absorption rate are presented.(3) After absorbing water, the gel does not flow to maintain the absorbent fiber strength and integrity of the original.(4) The manufacturing process of health supplies is simplified. Compared to resin, super absorbent fiber has wider applications. According to materials of high absorbent fibers, they can be divided into: cellulose, poly carboxylic acid, polyacrylonitrile, PEG modification. In this study, semi-finished PAN fiber as raw materials were hydrolyzed. In order to save costs and reduce emissions of NaOH solution to the environment, the effect of bath ratio and the recycling of the bath on fiber water absorptivity were further investigated.

2. Experimental

2.1 Materials

The semi-finished polyacrylonitrile (PAN) fiber as raw material was obtained from Acrylic Factory of Qilu Petrochemical. NaOH and HCl were analytically pure,which were supplied from Tianjin Bodi Chemical Co., Ltd.

2.2 Measurement of water absorbency

The dried fiber has been immersed in distilled water for 30 min. To fully absorbing water, the weight of fiber is weighed after the natural drops of 30 minutes. ,

The saturated water absorbency Q of fiber is expressed as follows;

$$Q\ (g/g)=\frac{W_1\text{-}W_0}{W_0}$$

Where, Q is the saturated water absorbency, W_1 is the quality of the sample before absorbing water, W_2 is the quality of the sample after absorbing water.

2.3 Measurement of bath viscosity

Bath viscosity is obtained from the viscosity measuring instrument.

3. Results and discussion

3.1 Effect of bath ratio on water-absorbency

Fig.1 shows the effect of different bath ratio on water-absorbency at alkaline conditions, for example, hydrolyzing temperature keeps at 95℃, the mass fraction of NaOH is 15%, water treatment is 60 min.It can be seen from Fig1 that bath ratio has little effect on the water absorptivity of superabsorbent fiber obtained by hydrolyzing PAN precursor. However,when the bath ratio is lower than 10:1, the fibers will not be fully immersed in bath solution and shows heterogeneous changes in color in the hydrolysis process,which will lead to inadequate hydrolysis and affect the uniformity of fiber absorbent.

Insert Figure 1 Here

3.2 Effect of bath cycles on water-absorbency

Fig.2 shows the effect of the recycling of the bath on fiber water absorptivity at alkaline conditions, for example, hydrolyzing temperature keeps at 95℃, the mass fraction of NaOH is 15%, water treatment is 60 min.the bath ratio is 40:1.It can be seen from Fig2 that after the first hydrolysis is complete, fibers are removed from the bath and simply squeezed,then liquid Extrusion was sent back to bath.The loss of NaOH in alkali solution is about 70%. After a certain quality of additional sodium hydroxide, the second reaction is began……and so on. It can be seen from Fig.2 that the water absorptivity of the first three hydrolyzing fibers remains stable. The water absorptivity of the fourth hydrolyzing fibers is slightly lower.However, the water absorptivity of the fifth hydrolyzing fibers is significantly lower.This seems to result from that some debris on the fiber macromolecules immerse in alkaline solution when PAN precursor is hydrolyzed. When the concentration of organic matter reach a certain amount, the collision probability of cyano and sodium hydroxide will be hindered, so that the hydrolysis of fibers is affected.

Insert Figure 2 Here

3.3 Effect of bath cycles on the change of fiber weight and bath liquid viscosity

Fig.2 shows the effect of the bath cycles on the change of fiber weight before and after hydrolysis at alkaline conditions, for example, hydrolyzing temperature keeps at 95℃, the mass fraction of NaOH is 15%, water treatment is 60 min. the bath ratio is 40:1. It can be seen from Fig.3 that the changes of the first three hydrolyzing fiber weight remain stable. The change of the fourth hydrolyzing fibers weight begin to decline. Compared with Fig.2, the above results show that although the bath of sodium hydroxide concentration remain unchanged, the degree of hydrolysis of PAN fiber samples have been affected greatly. As the hydrolysis process is carried out, fiber samples are dissolved from outside to inside simultaneously and the fiber layer is damaged, so that fiber weight is reduced. This is because with the bath cycles increasing, the remaining hydrolyzate in bath become more and more, bath viscosity is increased and the swelling of the fiber is reduced significantly.

Insert Figure 3 Here

Fig.4 shows the effect of the bath cycles on bath liquid viscosity at alkaline conditions, for example, hydrolyzing temperature keeps at 95℃, the mass fraction of NaOH is 15%, water treatment is 60 min. the bath ratio is 40:1. It can be seen from Fig4 that bath liquid viscosity become larger and larger with the increasing of bath cycles. Some debris on the fiber macromolecules immerse in alkaline solution,so that bath liquid viscosity increase. More debris on the fiber macromolecules is gradually accumulated with the increasing of bath cycles, so bath liquid viscosity become larger and larger.

Insert Figure 4 Here

3.4 The consumption of NaOH after bath cycles at three times

5g PAN fibers are hydrolyzed firstly at alkaline conditions, for example, the bath volume is 200ml, hydrolyzing temperature keeps at 95℃, the mass fraction of NaOH is 15%, water treatment is 60 min. Fibers are removed from the bath and simply squeezed in 60 min, then liquid Extrusion was sent back to bath. By titration, the bath concentration of 4.412 mol/L decreased to 4.150mol/L.The bath volume of 200ml is reduced to 188ml because of the bath loss of fiber attachment and the process of fiber extrusion. According to titration results NaOH is added in bath to the concentration before hydrolyzing, then the second response will begin……and so on. The bath volume is 160ml after the third hydrolyzing. The bath is sticky because some debris on the fiber macromolecules immerse in alkaline solution. As the viscosity is too high, it is difficult to filter. After the bath is diluted 5 times,it is fitered to remove debris on the fiber macromolecules in bath. Now the concentration of the bath is 3.668 mol/L by titration. In summary, after bath cycles at three times, NaOH of the bath can be recovered about 70%.

Insert Table 1 Here

4. Conclusion

The bath ratio has little effect on the water absorptivity of superabsorbent fiber obtained by hydrolyzing PAN precursor. The bath can be recycled at most three times, and maxmium recovery rate of the alkali solution is about 70%.

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Table 1. Bath volume and concentrations when bath cycles

Times	Before hydrolyzing	The first time	The second time	The third time
V(ml)	200	188	173	160
C(mol/L)	4.412	4.150	3.898	3.668

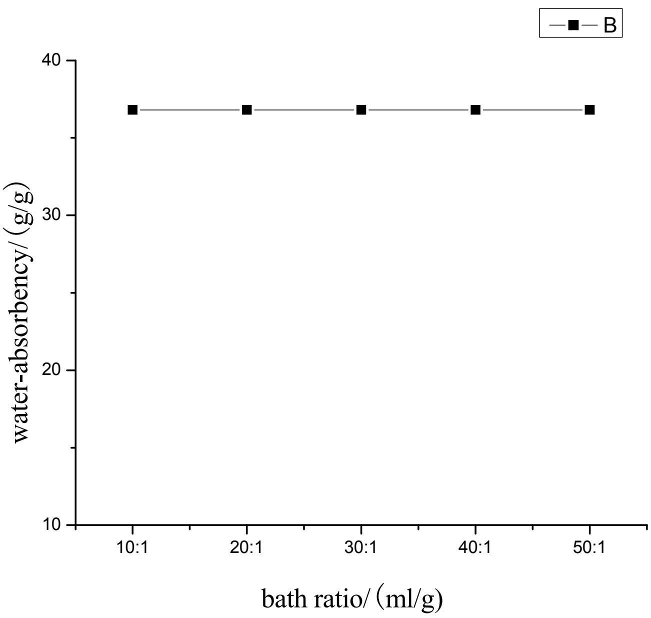

Figure 1. Effect of bath ratio on water-absorbency

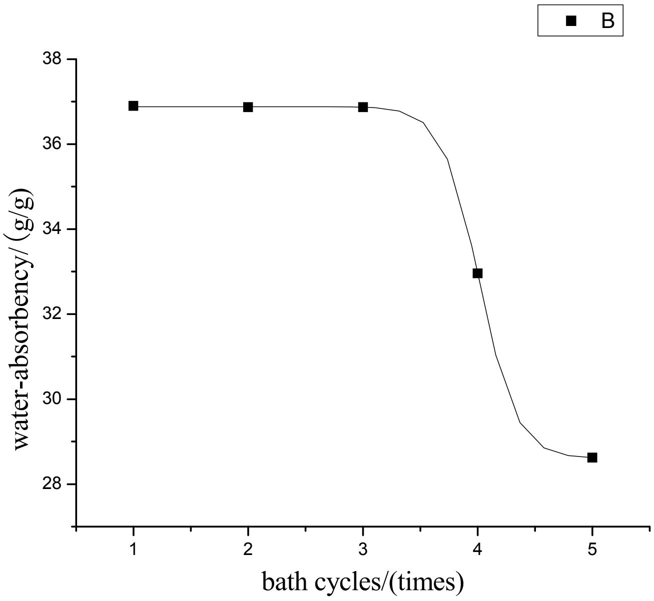

Figure 2. Effect of bath cycles on water-absorbency

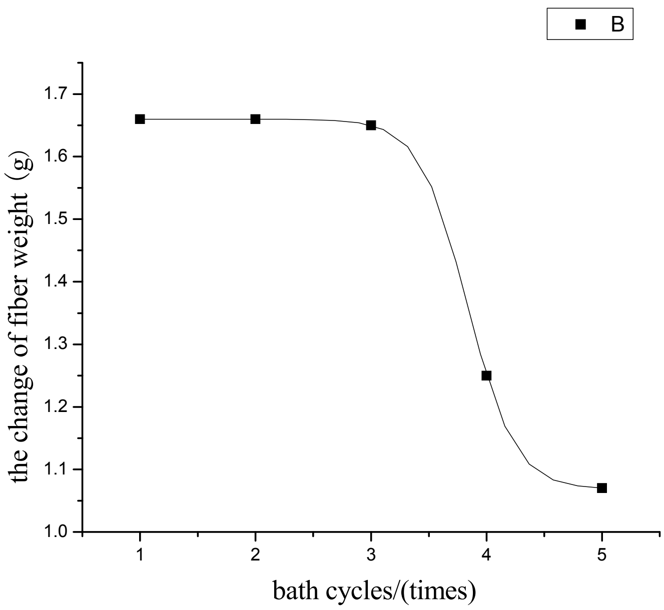

Figure 3. Effect of bath cycles on fiber weight

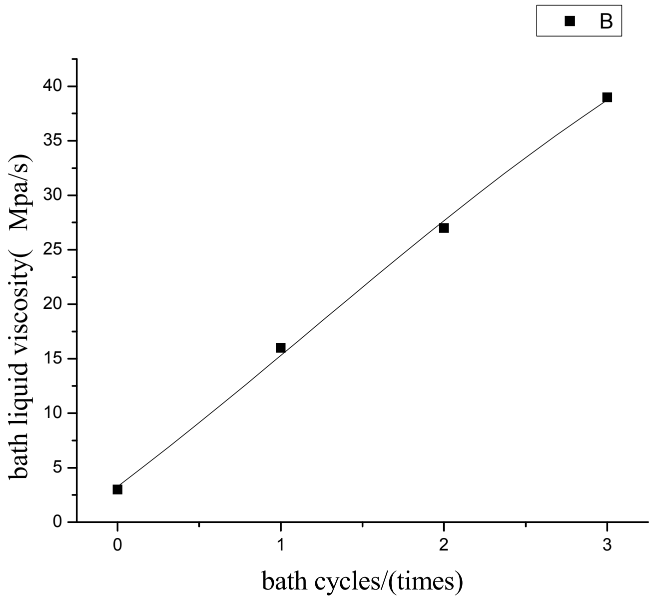

Figure 4. Effect of bath cycles on bath liquid viscosity