Study on Mechanical Properties and Micro Morphology of Acetate Filter Rod before and after Partial Degradation

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Abstract. Mechanical properties and micro morphology of acetate filter rod before and after partial degradation were studied. In this paper, the mechanical properties and micro morphology of acetate filter rod samples and 28 days soil burying were compared by means of Scanning Electronic Microscopy (SEM), texture analysis, infrared spectrum analysis and thermogravimetry (TG) analysis. The results showed that after degradation, the average compression modulus, hardness and elasticity of cellulose acetate filter rod decreased by 15.14%, 15.19% and 0.82%, respectively. The infrared spectrum analysis showed that cellulose acetate was degraded by soil burying method and carboxyl carbonyl was formed, but the degradation degree was not high. The degradation degree of cellulose acetate was lower than that of cellulose acetate before degradation. The weight loss zone between 110 ℃ and 230 ℃is less, which may be due to the complete degradation of the small molecular assistant added to the cellulose acetate degraded by the soil burying method.

1. Introduction
Acetate fiber is the second largest variety of cellulose fiber after viscose fiber. It has good physical and mechanical properties, and has the advantages of natural fiber (including man-made fiber) and synthetic fiber[1-3]. It is widely used in cigarette filter material, textile clothing, medical and health products and other industries[4]. As the filter material, diacetate fiber has the advantages of good filtration effect, moderate suction resistance, good thermal stability, non-toxic and tasteless, and the lower the fiber fineness, the better the filtration effect[5-7]. The cigarette filter rod made of acetate fiber can selectively absorb the harmful substances in the cigarettes. It can effectively absorb the tar and harmful chemicals in the cigarette without much changing the taste of the cigarette. Compared with other filter media, it provides the best balance between taste and price[8-10]. The environmental factors affecting the degradation of cellulose acetate include degradation temperature, environmental humidity, degradation medium, degradation time, pH value and oxygen content.

2. Experimental
The cellulose acetate filter rod was cut into a small section with a diameter of 7 mm and a length of 30 mm, and then dried in a vacuum drying oven at 25 ℃ for 24 hours, and divided into control sample and degradation sample.
JSM6390LV scanning electron microscope (SEM) of Japan Electronics Co., Ltd. was used to observe the micro morphology of the filter rod before and after degradation, and to investigate the damage of the micro structure of the filter rod caused by biodegradation. The effect of biodegradation on the mechanical properties of filter rod material was studied by texture analysis (TPA). The parameters are set as follows: test speed is 60 mm/min, compression ratio is 30%, minimum force is 1N, and trigger type is automatic. The filter rods before and after degradation were dissolved in acetone solution to prepare the membrane. The samples were scanned by attenuated total reflection (ATR) method and nexus 670 Fourier infrared spectrometer of Nicolet company in the United States.

The thermal stability of the samples was studied by TG 209 thermogravimetric analyzer of Netzsch company in Germany. Nitrogen flow rate is 20 ml / min, the temperature was increased at the rate of 10 °C / min, and the weight loss curve was recorded.

3. Results and discussion

3.1. Micro morphology observation

According to the results of the orthogonal test of the biodegradation rate of cellulose acetate filter rod, the c-ca filter rod degraded under the best degradation condition is selected for scanning electron microscopy observation, as shown in Fig. 1. As can be seen, the surface of the fiber bundles of filter rod without degradation is smooth and flat, and the shape of the incision is regular. However, after 28 days of degradation, the fiber bundles of the filter rod become slightly rough, and the difference of the incision section is slightly large, but the overall morphology is consistent with that of the filter rod without degradation. It can be seen that the microstructure of the filter rod is changed due to degradation, but the degradation is reduced. The degree of solution is not high.

![Fig. 1 cross section (a) and longitudinal plane (b) of cellulose acetate filter rod before degradation and cross section (c) and longitudinal plane (d) of cellulose acetate filter rod after degradation by SEM](image)

3.2. Mechanical properties

Mechanical properties of cellulose acetate filter rod before and after degradation were studied by texture analysis, as shown in Fig. 2. From the stress-strain curve of Fig. 1.a, it can be seen before degradation, the average compression modulus of cellulose acetate filter rod was 24.77 kPa, after degradation, the
average compression modulus of cellulose acetate filter rod changed to 21.02 kPa, decreased by 15.14%; before degradation, the average hardness of cellulose acetate filter rod was 7.57 N, after degradation, the average hardness of cellulose acetate filter rod changed to 6.42 N, decreased by 15.19%; before degradation, the average hardness of cellulose acetate filter rod changed to 6.42 N, decreased by 15.19% The average elasticity of cellulose filter rod is 0.979. After degradation, the average elasticity of cellulose acetate filter rod changes to 0.971, decreased by 0.82%, almost unchanged.

| Sample      | Compression modulus (kPa) | Hardness (N) | Elasticity |
|-------------|---------------------------|--------------|------------|
| Before-1    | 47.25                     | 7.51         | 0.995      |
| Before-2    | 18.94                     | 7.29         | 0.988      |
| Before-3    | 15.4                      | 7.74         | 0.969      |
| Before-4    | 17.50                     | 7.73         | 0.965      |
| Before-Average | 24.77                 | 7.57         | 0.979      |
| Before -SD  | 15.05                     | 0.21         | 0.016      |
| After-1     | 22.42                     | 6.34         | 0.958      |
| After-2     | 20.22                     | 5.72         | 0.973      |
| After-3     | 23.08                     | 6.53         | 0.955      |
| After-4     | 18.35                     | 7.07         | 0.996      |
| After-Average | 21.02                 | 6.42         | 0.971      |
| After-Sd    | 2.16                      | 0.55         | 0.019      |

3.3. Infrared spectrum analysis
Fig. 2.a and Fig. 2.b are infrared spectra of cellulose acetate before and after degradation, respectively. The sharp peak at 3328 cm\(^{-1}\) in Fig. 2.a may be the stretching vibration peak of \(-\text{O-H}\) bond in a small amount of unesterified carboxyl group in cellulose acetate molecule, while in Fig. 2.b there is a wide absorption peak in 3200 ~ 3600 cm\(^{-1}\), which may be due to the partial degradation of cellulose acetate molecule releasing part of carboxyl group and hydroxyl group, and hydrogen bond occurs between these groups, forming \(-\text{O-H-O-}\) bond The double frequency and combination frequency of stretching vibration and deformation vibration together form a broad blunt peak, but the peak strength is not large, indicating that cellulose acetate molecular degradation is not high, and does not release a large number of carboxyl and hydroxyl groups. In addition, compared with Fig. 2.a, the new absorption peaks in Fig. 2.b at 1734, 1366 and 1217 cm\(^{-1}\) are respectively the stretching vibration peaks of \(-\text{C=O}\) bond in carboxyl group, the
deformation vibration peaks of -C-H bond in carboxyl α position (CHCOO-) and the single stretching vibration peaks of -C-O bond in carboxyl group.

3.4. Thermogravimetry (TG) analysis

Thermogravimetry can measure the functional relationship between the variation of mass or mass surplus ratio and temperature, which can carry out quantitative analysis. Through TG curve, the quality of water, dry sample, the speed of dehydration and the rate of dehydration can be obtained. It is an important means to evaluate the thermal stability of fiber. Fig. 3 is the thermogravimetric analysis diagram of cellulose acetate filter rod before (a) and after (b) degradation, in which, black is the TG curve of sample, red is the DTG curve of sample, and dotted line is the tangent line of curve.

![Infrared spectrum of acetate fiber filter rod](image)

Fig. 3 Infrared spectrum of acetate fiber filter rod (a) before degradation and (b) after degradation

It can be seen from the TG curve of Fig. 3.a that the thermal weightlessness of cellulose acetate before degradation undergoes four processes: in the first step, under 110 °C, the weightlessness is about 0.4%, which may be caused by the adsorbed water or residual solvent in the cellulose acetate filter rod. In the second step, the weight loss is about 10.0% at 110-230 °C, which may be caused by the decomposition of small molecular assistant added in cellulose acetate filter rod. In the third step, at 230-400 °C, the weight loss is about 63.6%. This part of the weight loss is the decomposition of the sample fiber, which is the main stage of the weight loss. The initial decomposition temperature of cellulose acetate is 305.6 °C, the maximum weight loss rate temperature is 353.8 °C, and the weight loss between them is about 67.2%. In the fourth step, the weight loss is about 4.3% at 400 °C ~ 600 °C, and the weight loss rate is 81.9% at 600 °C.

From the TG curve of Fig. 3.b, it can be seen that the thermal weight loss of cellulose acetate after degradation mainly goes through three processes: the first step, under 110 °C, the weight loss is about 0.1%, which may be caused by the adsorbed water or residual solvent in the fiber. In the second step, the weight loss is about 82.5% at 110-420 °C, which is the main stage of the decomposition of the sample fiber. The initial decomposition of cellulose acetate is 333.6 °C, the maximum weight loss rate is 372.2 °C, and the weight loss between them is about 81.0%. In the third step, the weight loss is about 3.5% at 420- 600 °C, and the weight loss rate is 86.1% at 600 °C.
According to the above thermogravimetric analysis results, the weight loss area of the degraded cellulose acetate is less than that before degradation, which is between 110-230 ℃, which may be due to the complete degradation of the small molecular aids added in the cellulose acetate degraded by the soil burial method. In addition, the initial decomposition temperature, the maximum weight loss rate temperature and the weight loss rate from the initial decomposition temperature to the maximum weight loss rate temperature of the degraded cellulose acetate are higher than those of the degraded cellulose acetate, which may be due to the decomposition weight loss of some ester bonds exposed to the surface of the cellulose acetate tow after the degradation by the soil burial method. Due to the close aggregation of the sub chain due to the hydrogen bond, the decomposition temperature is slightly higher, and the molecular chain is loose after the degradation by the soil burial method, the degradation proportion of the main body is increased compared with that before the degradation, and the final weight loss rate is increased by 4.2% compared with that before the degradation.

4. Conclusion
(1) After 28 days of soil burial degradation, under the optimal degradation condition, the microstructure of the filter rod changed because of degradation, but the degradation degree was not high. The mean value of compression modulus, hardness and elasticity of cellulose acetate filter rod decreased by 15.14%, 15.19% and 0.82%, respectively.
(2) The weight loss area of cellulose acetate after degradation is less than that before degradation, which is between 110-230 ℃. It may be due to the complete degradation of small molecular aids added in cellulose acetate after soil burying method. The degradation proportion of the main body is increased compared with that before the degradation, and the final weight loss rate is increased by 4.2% compared with that before the degradation.

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