

Optimization of Technological Parameters for Preparation of Vegetable Fiber Mulch Film with Waste Corrugated Paperboard

Xianglan Ming, Haitao Chen

School of Engineering, Northeast Agricultural University, Harbin 150030, China

E-mail: 792146915@qq.com, htchen@neau.edu.cn

Abstract: The process and properties of vegetable fiber film made from waste corrugated paperboard were studied in order to solve the pollution problem of traditional plastic film to soil and environment and efficiently reuse of waste corrugated paperboard. Experiments were conducted using waste corrugated cardboard and rice straw as the main raw materials, adding environmental protection type functional auxiliaries, with the clean pulping and papermaking process to prepare biodegradable vegetable fiber mulch film. Study on the effects of technological parameters on dry tensile strength, wet tensile strength and sizing value, the optimization of the technological parameters effect on the mulching made from waste corrugated cardboard was studied by the method of five factors and five levels 1/2 orthogonal rotating center combination test method. The five technological parameters which are considered in this study for optimization are basis weight, neutral sizing agent, ratio, wet strength agent and beating degree. Experimental results indicated that an optimum combination is basis weight 56 to 75 g/m², ratio 0% to 16%, neutral sizing agent 1.6%, wet strength agent 2.4%, beating degree 30 °SR, in this case, dry tensile strength of this fiber film attained more than 40 N, wet tensile strength more than 15 N and sizing value more than 120 s, and the film met the requirements of mechanical property for field mulching. The results provided theoretical basis and technical support for the preparation of degradable fiber mulch film from waste corrugated paperboard.

Key words: Waste corrugated paperboard; Rice straw; Biodegradable mulch film; Orthogonal rotating center combination test; Mechanical strength

1. Introduction

China is a large agricultural country. In the past thirty years, agricultural films have been developing rapidly in China's agricultural production. The output of agricultural plastic film has been ranked the first in the world, 1.6 times the sum of all other countries [1]. In the past ten years, the annual output of plastic film in China has reached 1 million tons, covering more than 20 million hm². The plastic film mulching technology has been popularized in the whole country [2]. But each year soil residual film was remained in the soil because of the great cost of recycling, causing soil pollution, destroying soil physical and chemical structure, leading to negative effects of agricultural production reduction, emerging environmental crisis, and threatening the ecological security of China's agricultural population [3-5]. Therefore, in order to solve the problem of soil pollution of plastic mulch film accumulated in the farmland soil, it is imperative to study the biodegradable vegetable fiber mulch film.

Corrugated paperboard is made of wood fiber, which can be decomposed by natural function, which is a great contribution to the treatment of packaging waste. In the absence of government regulation, the degree of recycling is determined by the economy of each material, and corrugated paperboard is no exception [6-8]. Based on the economy of corrugated paperboard and the strong recycling, the waste corrugated paperboard can be recycled by 100% in theory, but in fact the degree of recycling is not high. From the existing public data, there is no accurate data on the current utilization rate of corrugated paperboard in China [9-11]. But from the data released by some companies, the utilization rate has been rising. This indirectly reflects that the economic benefits reflected by the recycle utilization of corrugated paperboard can promote the enterprises to improve its recycling efficiency.

Waste corrugated paperboard is widely used as papermaking raw material as an important renewable resource for papermaking industry, not only can ease the shortage of papermaking raw material and energy consumption of the contradiction, but also can accord with the concept of sustainable development and alleviate the increasingly serious environmental pollution problems [12]. However, in the process of recycling, a series of physical processes such as crystallization and irreversible closure of pores of the corrugated paperboard will lead to the deterioration of the fiber properties. Therefore, it is of great significance to study the influence of recycled corrugated paperboard on the performance of fiber mulch film in order to enhance the performance of fiber mulch film and improve the utilization efficiency of waste corrugated fiberboard[13-16].

The purpose of this study is to optimize the combination of waste corrugated paperboard and rice straw fiber to make biodegradable fiber film through orthogonal rotating center combination test, in order to explore the feasibility of using waste corrugated paperboard and rice straw to make biodegradable fiber film, and to provide theoretical basis for making degradable fiber film from waste corrugated paperboard.

2. Materials and methods

2.1. Materials

The 425 rice straw was provided by Northeast Agricultural University in the fall of 2016 (less than 10 mm fiber was retained through the analysis of FB-1 type sieve and the average aspect ratio was in the range of 22.8~58.3); Waste corrugated paperboard (avoiding the printing part) was taken from a domestic Paper Co; Wet strength agent was provided by Xinghuo Chemical Plant (Mudanjiang, China); Neutral sizing agent was provided by Jinhao Chemical Plant (Qingzhou, China); Iron trichloride was provided by Tianli Chemical Reagent Co., Ltd.(Tianjin, China); Ammonium thiocyanate was provided by Fuchen Chemical Auxiliaries Factory(Tianjin, China).

2.2. Equipment and instrument

D200 type of straw fiber extruder, manufactured by Northeast Agricultural University; electronic scale, Haikang electronic instrument factory (Shanghai, China); ZT-400 Valli beater, Zhongtong Test Equipment Co., Ltd (Shanxi, China); JA5003B electronic balance, Tianmei science and technology instrument Co., Ltd (Shanghai, China); ZTG-100 pulp degree test machine, ZCX-A paper sheet forming device, ZL-300 pendulum paper tension strength test machine, ZDNP-1 paper bursting strength test machine, ZUS-4 paper thickness measuring instrument, Yueming small test machine Co., Ltd (Changchun, China); Plus V3.5.0 *Leica Qwin* optical microscopy (Made in Germany).

2.3. Methods

Five factors and five levels 1/2 orthogonal rotating center combination test method was employed for investigation the effects of process parameters on dry tensile strength, wet tensile strength and sizing value. According to the literature survey and experimental observations, the five process parameters which were considered to influence the performance of waste corrugated paperboard and rice straw blended fiber film in this study for optimization were basis weight, neutral sizing agent, ratio, wet strength agent and beating degree. All the indexes were tested by the mean of ten repetitions. Ratio refers to the percentage of dry matter in rice straw fiber to total pulp. Tensile strength is related to the fiber properties, fiber bonding strength and fiber arrangement. Among them, the size and nature of the fiber bonding force are the most important conditions affecting the effective tensile strength. Sizing refers to the sizing of the film to obtain resistance to fluid penetration and diffusion, and the sizing value is an important indicator to evaluate the hydrophobic properties of the film [17-20]. In this test, the beating degree of waste corrugated paperboard was maintained constant at 45 °SR for all experiments. For each process parameter, five levels were chosen as shown in Table 1.

Table 1 Factors level code

Level	Factors				
	Basis weight $x_1/(g/m^2)$	Neutral sizing agent $x_2/\%$	Ratio $x_3/\%$	Wet strength agent $x_4/\%$	Beating degree $x_5/ (^\circ\text{SR})$
-2	55	1.5	0	1.8	30
-1	60	1.6	15	2.0	35
0	65	1.7	30	2.2	40
1	70	1.8	45	2.4	45
2	75	1.9	60	2.6	50

2.4. Mechanical properties

Mechanical tests were carried out in the Biomass Laboratory of Northeast Agricultural University, China. Mulch film strips (15 mm wide) were blade-cut in machine directions and stored at 20 °C and 50% RH for one week prior to testing. Tensile tests were performed on a ZL-300 pendulum paper tension strength test machine (China) using a 0.5 kN force sensor and a 120 mm sample gauge lengths. Tests were conducted in ten repetitions according to GB/T 453-1989 standard and GB/T 465.2-1989 standard at 150 mm/min loading speed, so expressing the results in terms of average value.

Liquid permeance method is the determination of the sizing value based on the time required for the liquid to

pass through the paper. The sizing value of the sample was determined by GB/T 5405-1985 which was indicated the average time value of the positive and negative test results in ten repetitions. 30 × 30 mm specimen was used to make a boat about 20 × 20 mm. In the culture dish, pour in a proper amount of 20 g/L solution of ammonium thiocyanate, and place the boat on the surface of the water. At the same time, drop 1 drops of 10 g/L solution of ferric chloride into the boat with a dropper. Start the stopwatch and stop the stopwatch when the red dot appeared.

The results obtained from tensile tests are reported in terms of dry tension strength (σ_d) and wet tension strength (σ_w) expressed in [N], sizing value (ϵ_s) expressed in [s].

2.5. Preparation of vegetable fiber mulch film with waste corrugated paperboard

1) Cut the 425 rice straw harvested in autumn of 2016 into 100mm, soak in normal atmospheric temperature for 12h. Then use D200 type of straw fiber extruder produce coarse fiber.

2) Beating rice straw fiber and waste corrugated paperboard to the corresponding beating degree of the test scheme referring to GB/T 3702-1999.

3) Mix the two kind pulps together according to the proportions in the test program, calculate the additive amount of every additive and then stir the mixing pulp evenly [21].

4) Referring to GB/T 3703-1999, use the well stirred pulp making film samples with the corresponding basis weight in the scheme. Then put film samples on the drying machine to be dried completely (vacuum degree up to 96 kPa, temperature approximately of 97 °C and drying time of 5 ~ 7 minutes). Samples were allowed to dry at room temperature (23±1 C temperature and 50%±2% relative humidity) for 24h before measurement.

5) Refer GB/T 453-1989 and GB/T 465.2-1989 to measure the dry, wet tension strength, refer GB/T 5405-1985 to measure sizing value of film samples. Every experiment was taken for ten times. Use Design-Expert software to make statistical analysis of the data.

3. Results and analysis

3.1. Test result

The test results were shown in Table 2.

Table 2 Experimental plan and results

No.	Basis weight	Neutral sizing agent	Ratio	Wet strength agent	Beating degree	Dry tensile strength/N	Wet tensile strength/N	Sizing degree/(s)
1	-1	-1	-1	-1	1	30.8	9.6	84
2	1	-1	-1	-1	-1	40.8	11.4	90
3	-1	1	-1	-1	-1	21.2	7.3	62
4	1	1	-1	-1	1	39.9	11.5	102
5	-1	-1	1	-1	-1	20.4	5.6	28
6	1	-1	1	-1	1	23.2	5.9	40
7	-1	1	1	-1	1	23.7	6.7	33
8	1	1	1	-1	-1	28.4	8.7	98
9	-1	-1	-1	1	-1	36.5	13.5	105
10	1	-1	-1	1	1	39.0	13.3	130
11	-1	1	-1	1	1	33.8	10.9	97
12	1	1	-1	1	-1	40.0	14.8	134
13	-1	-1	1	1	1	20.3	7.3	84
14	1	-1	1	1	-1	24.5	6.7	72
15	-1	1	1	1	-1	26.7	7.8	102
16	1	1	1	1	1	27.2	8.1	112
17	-2	0	0	0	0	25.2	7.1	65
18	2	0	0	0	0	38.7	11.9	137
19	0	-2	0	0	0	30.2	10.8	79
20	0	2	0	0	0	29.4	9.6	104

21	0	0	-2	0	0	46.8	17.5	151
22	0	0	2	0	0	14.3	3.9	28
23	0	0	0	-2	0	27.1	7.8	66
24	0	0	0	2	0	36.1	11.1	117
25	0	0	0	0	-2	34.7	11.0	123
26	0	0	0	0	2	31.4	9.3	137
27	0	0	0	0	0	34.5	8.7	132
28	0	0	0	0	0	33.5	9.2	108
29	0	0	0	0	0	33.4	7.9	110
30	0	0	0	0	0	31.4	8.7	98
31	0	0	0	0	0	30.4	9.0	91
32	0	0	0	0	0	30.0	11.0	134
33	0	0	0	0	0	28.9	9.0	91
34	0	0	0	0	0	29.1	9.6	121
35	0	0	0	0	0	31.7	9.7	87
36	0	0	0	0	0	34.9	9.1	108

3.2. Test analysis

3.2.1. Effects of technological parameters on dry tensile strength

The influence of technological parameters such as basis weight, neutral sizing agent, ratio, wet strength agent and beating degree on dry tensile strength of biodegradable mulch film had been investigated by employing Central Composite Design and response surface methodology. All main effects and two factor interaction effects as obtained by ANOVA was shown in Table 3.

Table 3 Variance analysis of regression model

Source	Sum of Squares	DF	Mean Square	F Value	Prob>F	Critical value
Model	1404.11	8	175.51	$F_2=23.61$	<0.0001	$F_{0.05}(8 \cdot 27)=2.31$
x_1	244.48	1	244.48	32.89	<0.0001	
x_2	0.60	1	0.60	0.081	0.7782	
x_3	970.28	1	970.28	130.52	<0.0001	
x_4	58.91	1	58.91	7.92	0.0090	
x_5	2.16	1	2.16	0.29	0.5943	
x_1x_3	39.69	1	39.69	5.34	0.0287	
x_1x_4	32.49	1	32.49	4.37	0.0461	
x_2x_3	55.50	1	55.50	7.47	0.0110	
Residual	200.72	27	7.43			
Lack of fit	157.31	18	8.74	$F_1=1.81$	0.1822	$F_{0.05}(18 \cdot 9)=2.968$
Pure error	43.42	9	4.82			
Total	1604.84	35				

The Model F -value of 23.61 implies the model was significant. There was only a 0.01% chance that a "Model F -Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms were significant. In this case x_1 , x_3 , x_4 , x_1x_3 , x_1x_4 , x_2x_3 were significant model terms. The "Lack of Fit F -value" of 1.81 implies the Lack of Fit was not significant relative to the pure error. There was only a 18.22% chance that a "Lack of Fit F -value" this large could occur due to noise. Final Equation in Terms of Coded Factors was:

$$y_1 = 30.78 + 3.19x_1 + 0.16x_2 - 6.36x_3 + 1.57x_4 - 0.30x_5 - 1.58x_1x_3 - 1.43x_1x_4 + 1.86x_2x_3 \quad (1)$$

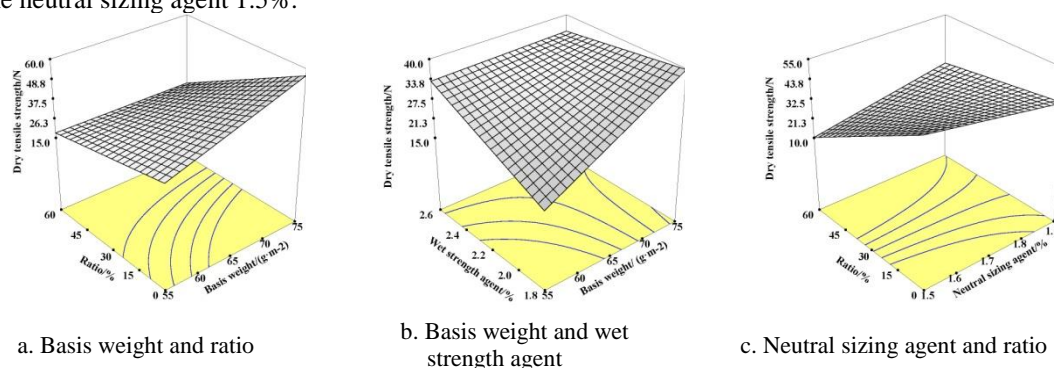
where, x_1 , x_2 , x_3 , x_4 , and x_5 indicated basis weight(g/m²), neutral sizing agent(%), ratio(%), wet strength agent(%) and beating degree(°SR), respectively.

This equation could well predict the theoretical dry tensile strength of biodegradable mulch film with known technological parameters.

Combined effect of basis weight and ratio on dry tensile strength of biodegradable mulch film at other parameters (0 levels) had been shown in Fig. 1a. Below 0 levels of ratio, the dry tensile strength was positively related to the basis weight, and with the increase of the basis weight, the dry tensile strength gradually increased. This was mainly because the strength of the sample was proportional to the number of fibers on the sample per unit area as increasing the amount of the total fiber on the film sample per unit area was beneficial to increase the bonding force between the fibers, and finally increase the dry tensile strength of the fiber; The change of dry tensile strength was not obvious with the increase of basis weight above 0 levels of ratio. With the increase of ratio, the proportion of rice straw fiber in unit film area was increased, and even if the basis weight increases, dry tensile strength had no obvious trend of change. According to the response surface Fig. 1a, the effect of basis weight and ratio on dry tensile strength was the same. The maximum value appeared in the basis weight 75 g/m² and the ratio 0%.

Combined effect of basis weight and wet strength agent on dry tensile strength of biodegradable mulch film at other parameters (0 levels) had been shown in Fig. 1b. It could be seen that below 0 levels of basis weight as wet strength agent increased, dry tensile strength of the sample increased. This was due to the wet strength agent in the film could not only covalently bound to the carboxyl group of the fiber, but also form water insoluble and network structures between molecules or molecules via self bridging reactions in the wet strength agent, which had a significant enhancement effect; Above 0 levels of basis weight the dry tensile strength decreased with the increase of the wet strength agent, but the range was small. This might be because wet strength agent provided cationic charge, and fiber bands were anionic charges. With the increasing of the basis weight, the anion charge increased with the increase of the anion charge in the film. In the system, wet strength agent no longer played a role in increasing the dry tensile strength with the saturation adsorption of fiber on the wet strength agent. According to the response surface Fig. 1b, the effect of basis weight on dry tensile strength was slightly higher than that of wet strength agent, and the maximum value was basis weight 75 g/m², and the wet strength agent 1.8%.

Combined effect of neutral sizing agent and ratio on dry tensile strength of biodegradable mulch film at other parameters (0 levels) had been shown in Fig. 1c. The dry tensile strength decreased with the increase of the neutral sizing agent below 0 levels of ratio. It was mainly due to cationic neutral sizing agent adding to the slurry, and then formed physical adsorption between the negatively charged slurry fibers. The presence of medium low degree component (or discrete fragments) in neutral sizing agents resulted in uneven distribution of fiber surface charges and local cationic patches; Above 0 levels of ratio as neutral sizing agent increased, dry tensile strength of the sample increased. This was because the neutral sizing agent was esterified with cellulose, and the gum was adsorbed on the surface of the film to form a lot of hydrogen bonding points, thus increasing the bonding force between the slurry fibers [22]. According to the response surface Fig. 1c, the effect of ratio on dry tensile strength was much greater than that of neutral sizing agent. The maximum value appeared in the ratio 0% and the neutral sizing agent 1.5%.



Note: Other technological parameters were fixed at 0 levels

Fig.1 Response surface for the effects of technological parameters on dry tensile strength

3.2.2. Effects of technological parameters on wet tensile strength

The influence of technological parameters such as basis weight, neutral sizing agent, ratio, wet strength agent and beating degree on wet tensile strength of biodegradable mulch film had been investigated by employing Central Composite Design and response surface methodology. All main effects and two factor interaction effects as obtained by ANOVA was shown in Table 4.

The Model *F*-value of 25.56 implies the model was significant. There was only a 0.01% chance that a "Model *F*-Value" this large could occur due to noise. Values of "Prob > *F*" less than 0.0500 indicate model terms were significant. In this case x_1 , x_3 , x_4 , x_1x_2 , x_2x_3 , x_3x_4 were significant model terms. The "Lack of Fit *F*-value" of 1.96 implies the Lack of Fit was not significant relative to the pure error. There was only a 15.11% chance that a

"Lack of Fit F-value" this large could occur due to noise. Final Equation in Terms of Coded Factors is:

$$y_2 = 9.47 + 0.89x_1 + 4.167E^{-003}x_2 - 2.61x_3 + 0.93x_4 - 0.25x_5 + 0.57x_1x_2 + 0.57x_2x_3 - 0.61x_3x_4 \quad (2)$$

where, x_1 , x_2 , x_3 , x_4 , and x_5 indicated basis weight(g/m²), neutral sizing agent(%), ratio(%), wet strength agent(%) and beating degree(°SR), respectively.

Table 4 Variance analysis of regression model

Source	Sum of Squares	DF	Mean Square	F Value	Prob>F	Critical value
Model	221.11	8	27.64	$F_2=25.56$	<0.0001	$F_{0.05}(8 \cdot 27) = 2.31$
x_1	18.90	1	18.90	17.48	0.0003	
x_2	4.167E ⁻⁰⁰⁴	1	4.167E ⁻⁰⁰⁴	3.853E ⁻⁰⁰⁴	0.9845	
x_3	163.80	1	163.80	151.45	<0.0001	
x_4	20.72	1	20.72	19.16	0.0002	
x_5	1.45	1	1.45	1.34	0.2570	
x_1x_2	5.18	1	5.18	4.79	0.0375	
x_2x_3	5.18	1	5.18	4.79	0.0375	
x_3x_4	5.88	1	5.88	5.44	0.0274	
Residual	29.20	27	1.08			
Lack of fit	23.27	18	1.29	$F_1=1.96$	0.1511	$F_{0.05}(18 \cdot 9) = 2.968$
Pure error	5.93	9	0.66			
Total	250.31	35				

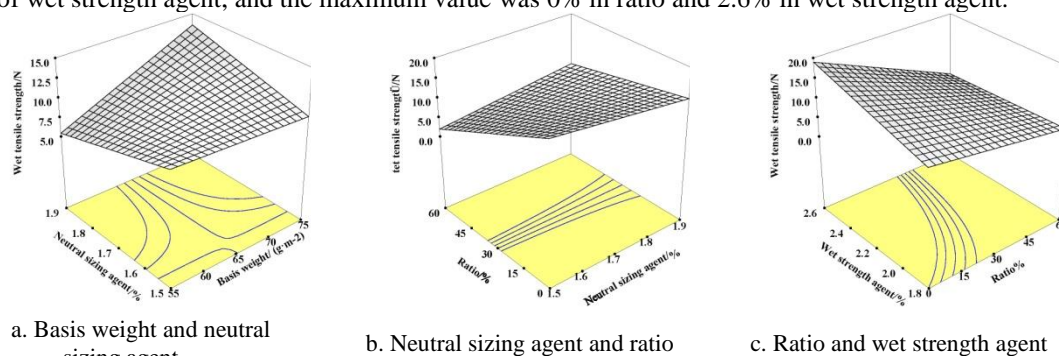
This equation could well predict the theoretical wet tensile strength of biodegradable mulch film with known technological parameters.

Combined effect of basis weight and neutral sizing agent on wet tensile strength of biodegradable mulch film at other parameters (0 levels) had been shown in Fig. 2a. When the basis weight and neutral sizing agent was below 0 levels, the wet tensile strength decreased with the increase of the basis weight and neutral sizing agent; above 0 levels of basis weight and neutral sizing agent as basis weight and neutral sizing agent increased, wet tensile strength of the sample increased. This was because the neutral sizing agent readily reacted with fibers to form ketene ester derivatives, which was localized on the fiber and forms a stable film on the surface of the fiber. At this time, the hydrophobic group (long chain alkyl) shifted to the surface of the fiber, which made the film obtain hydrophobic properties, and the wet tensile strength increased continuously. According to the response surface Fig. 2a, the influence of basis weight on wet tensile strength was much higher than that of neutral sizing agent. The maximum value appeared to be basis weight 75 g/m² and the neutral sizing agent 1.9%.

Combined effect of neutral sizing agent and ratio on wet tensile strength of biodegradable mulch film at other parameters (0 levels) had been shown in Fig. 2b. The wet tensile strength decreased slowly with the increase of the neutral sizing agent below 0 levels of ratio. The main reason was that when the amount of neutral sizing agent was too large, the emulsion particles became larger, the stability decreased, and the dispersion in water became worse. In the same amount, the number of colloidal particles decreased, the total surface area of colloidal particles decreased, and the contact area of fibers decreased correspondingly, the retention of rubber decreased rapidly, the effect of sizing decreased, and the wet tensile strength decreased accordingly; The wet tensile strength increased with the increase of the neutral sizing agent above 0 levels of ratio. This was because of the sizing agent sizing effect, the primary fiber paste was superior to the two (multiple) fiber paste, and the reuse of waste corrugated paperboard belonged to the two fiber sizing agent. Therefore, when the ratio was large, the sizing effect of the neutral sizing agent on the rice straw fiber (primary fiber sizing agent) was enhanced, and the wet tensile strength was increased. According to the response surface Fig. 2b, the effect of ratio on the wet tensile strength was much greater than that of the neutral sizing agent. The maximum value was 0% in the ratio and 1.5% in the neutral sizing agent.

Combined effect of ratio and wet strength agent on wet tensile strength of biodegradable mulch film at other parameters (0 levels) had been shown in Fig. 2c. The wet tensile strength increased with the addition of wet strength agent below 0 levels of ratio. Mainly in the process of recycling, the lignin of waste corrugated paperboard would be colloid, which was strengthened the bonding force between the slurry fibers. At the same time, wet strength agent was a kind of low molecular weight water soluble resin. When it was added to the slurry, it would penetrate into the surface and interior of the fiber, and effectively crossed link with the fiber, so that the wet tensile strength increased; the wet tensile strength decreased slightly with the increase of the wet strength agent above 0 levels of ratio. This was because the wet strength agent was consumed by the use of neutral sizing agent, and the probability of combining with the fiber into the film was reduced, thus affecting the wet tensile

strength. According to the response surface Fig. 2c, the effect of ratio on wet tensile strength was slightly higher than that of wet strength agent, and the maximum value was 0% in ratio and 2.6% in wet strength agent.



Note: Other technological parameters were fixed at 0 levels

Fig.2 Response surface for the effects of technological parameters on wet tensile strength

3.2.3. Effects of technological parameters on sizing value

The influence of technological parameters such as basis weight, neutral sizing agent, ratio, wet strength agent and beating degree on sizing value of biodegradable mulch film had been investigated by employing Central Composite Design and response surface methodology. All main effects and two factor interaction effects as obtained by ANOVA was shown in Table 5.

Table 5 Variance analysis of regression model

Source	Sum of Squares	DF	Mean Square	F Value	Prob>F	Critical value
Model	27914.09	10	2791.41	$F_2=9.61$	<0.0001	$F_{0.05}(10 \cdot 25) = 2.24$
x_1	4455.38	1	4455.38	15.33	0.0006	
x_2	1027.04	1	1027.04	3.53	0.0718	
x_3	9640.04	1	9640.04	33.18	<0.0001	
x_4	6700.04	1	6700.04	23.06	<0.0001	
x_5	15.04	1	15.04	0.052	0.8219	
x_2^2	1270.92	1	1270.92	4.37	0.0468	
x_3^2	1480.59	1	1480.59	5.10	0.0330	
x_4^2	1270.92	1	1270.92	4.37	0.0468	
x_1x_2	915.06	1	915.06	3.15	0.0881	
x_2x_3	1139.06	1	1139.06	3.92	0.0588	
Residual	7263.46	25	290.54			
Lack of fit	4719.46	16	294.97	$F_1=1.04$	0.4940	$F_{0.05}(16 \cdot 9) = 2.99$
Pure error	2544.00	9	282.67			
Total	35177.56	35				

The Model F -value of 9.61 implies the model was significant. There was only a 0.01% chance that a "Model F -Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms were significant. In this case x_1 , x_3 , x_4 , x_2^2 , x_3^2 , x_4^2 were significant model terms. The "Lack of Fit F -value" of 1.04 implies the Lack of Fit was not significant relative to the pure error. There was only a 49.40% chance that a "Lack of Fit F -value" this large could occur due to noise. Final Equation in Terms of Coded Factors was:

$$y_3 = 109.05 + 13.63x_1 + 6.54x_2 - 20.04x_3 + 16.71x_4 + 0.79x_5 - 6.30x_2^2 - 6.80x_3^2 - 6.30x_4^2 + 7.56x_1x_2 + 8.44x_2x_3 \quad (3)$$

where, x_1 , x_2 , x_3 , x_4 , and x_5 indicated basis weight (g/m^2), neutral sizing agent (%), ratio (%), wet strength agent (%) and beating degree ($^\circ\text{SR}$), respectively.

This equation could well predict the theoretical sizing value of biodegradable mulch film with known technological parameters.

Combined effect of basis weight and neutral sizing agent on sizing value of biodegradable mulch film at other parameters (0 levels) had been shown in Fig. 3a. The sizing value increased first and then decreased with the increase of the neutral sizing agent below 0 levels of basis weight. This was because the traditional cationic neutral sizing agent was mainly retained on the surface of the fine fibers of the slurry. In general, the cationic component in the emulsion and anionic fibers attracted positive and negative charge, and then stayed in the film through the retention agent; the sizing value gradually increased with the increase of the neutral sizing agent

above 0 levels of basis weight. This might be because, as the basis weight increases continuously, the total number of fibers on the film sample per unit area increased. Neutral sizing agents readily reacted with fibers at high temperatures in the drying section to form ketene ester derivatives and increased sizing value. According to the response surface Fig. 3a, the influence of basis weight on sizing value was much greater than that of neutral sizing agent, and the maximum value was basis weight 75 g/m² and the neutral sizing agent 1.9%.

Combined effect of neutral sizing agent and ratio on sizing value of biodegradable mulch film at other parameters (0 levels) had been shown in Fig. 3b. The sizing value increased first and then decreased with the increase of the neutral sizing agent below 0 levels of ratio. This might be due to the retention rate of the slurry system worse than that of the static state. As a result, the portion of the neutral sizing particles that should be left on the fine fraction was reduced to the white water due to a decrease in the retention rate of the fine fraction, and the sizing effect of the film was affected; the sizing value gradually increased with the increase of the neutral sizing agent above 0 levels of ratio. This might be because at this amount, a certain amount of epoxies groups in the wet strengthening agent ring open led secondary amine alkylation to nucleophilic reagent. It attacked the lactones ring of a certain amount of neutral sizing agent, and caused it to be opened and fixed on the macromolecular chain of wet strength resin, so as to play a synergistic effect [23]. According to the response surface Fig. 3b, the influence of ratio on sizing value was much greater than that of neutral sizing agent, and the maximum value was 0% in ratio and 1.7% in neutral sizing agent.

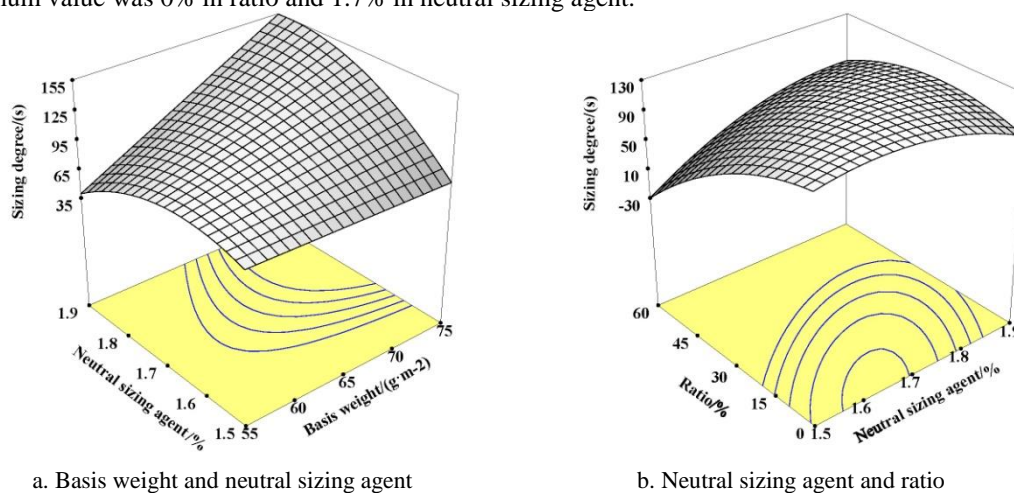


Fig.3 Response surface for the effects of technological parameters on sizing value

3.3. Optimization analysis

In order to meet the requirements of mechanical performance in the field of film mulching, namely the film mechanical strength in accordance with the national standard GB-13735-1992, the dry tensile strength more than 40 N, wet tensile strength greater than 15 N, the sizing value larger than 120 s, and to save energy and reduce costs as the principle. The optimum analysis result was shown in Fig. 4 when the basis weight 55 to 75 g/m², neutral sizing agent 1.5% to 1.9%, ratio 0% to 60%, wet strength agent 1.8% to 2.6%, and beating degree 30 to 50 °SR. The optimum combination of technological parameters was basis weight 56 to 75 g/m², ratio 0% to 16%, neutral sizing agent 1.6%, wet strength agent 2.4%, and beating degree 30 °SR, which could meet the objective function requirements.

3.4. Verification test

Manufacturing film samples was according to the optimal technological parameters, which were basis weight 70 g/m², ratio 10%, neutral sizing agent 1.6%, wet strength agent 2.4%, and beating degree 30 °SR. Evaluation indicators were separately determined and 10 parallel tests were taken for the verifications. Experimental results indicated that dry tensile strength was 47.4 N, wet tensile strength was 16.2 N and sizing value was 137 s, which showed that the optimization results were correct and credible.

3.5. Discussion

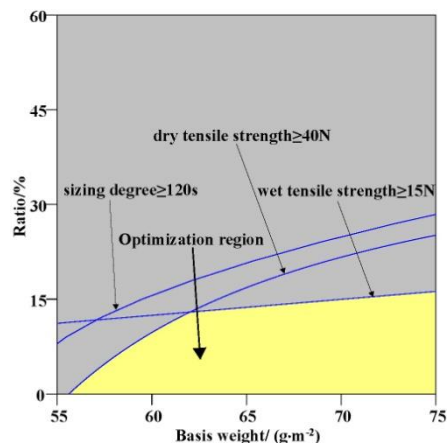
In this paper, biodegradable mulch film was prepared from rice straw fiber and waste corrugated paperboard two times fiber as raw material. Mainly for the technological parameters of the film mechanical properties and parameters of the combination of optimization research, but there were still some shortcomings:

1) The existing auxiliary ingredients in waste corrugated paperboard couldn't be clearly recovered, and more impurities were contained in the recycled fiber. A variety of fillers and additives couldn't be completely

separated. The residual of auxiliary agent had some influence on the properties of the recycled material.

2) The cost of making vegetable fiber film should meet the needs of the society as much as possible. Economy was an important factor to consider whether the material was feasible and whether the finished product could be popularized or not. In the future, it was necessary to study the assistant agent with obvious effect and economic and environmental protection.

3) An anionic garbage accumulation problem was caused by the white water closure.



Note: neutral sizing agent 1.6% · wet strength agent 2.4 % · beating degree 30°SR.

Fig.4 Optimum analysis of technological parameters

4. Conclusions

1) The rank of the effect of five factors on dry tensile strength from high to low was as follows: ratio, basis weight, wet strength agent, neutral sizing agent and beating degree; on wet tensile strength: ratio, wet strength agent, basis weight, neutral sizing agent and beating degree; on sizing value: ratio, wet strength agent, basis weight, neutral sizing agent and beating degree.

2) Optimal technology parameters of degradable fiber mulch film made from waste corrugated paperboard and rice straw were basis weight 56 to 75 g/m², ratio 0% to 16%, neutral sizing agent 1.6%, wet strength agent 2.4%, and beating degree 30 °SR; under this condition, dry tensile strength was greater than 40 N, wet tensile strength was greater than 15 N, sizing value was greater than 120 s, and the film met the requirements of mechanical property for field mulching.

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