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Study on Manufacturing Technology and Performance of Biogas Residue Film

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1. Introduction

Plastic mulching cultivation technology originated from Japan in 1955. This technology was introduced to China in 1978, and it was comprehensively spread after a series of tests. Plastic film mulching technology was widely used due to increasing temperature, preserving soil moisture, increasing yield, preventing soil erosion etc. To 1986, the using area of plastic film in China had leap to the first in the world. The application of mulching cultivation technology is an effective way to improve crop yield, and has promoted the development of agriculture. The application of plastic film was known as the third revolution following fertilizer, seeds in agriculture, also called "white revolution" (Zhang Ying, 2005).

However, plastic is a polymer material, which is non-perishable, difficult degradation, experiments show that the degradation of plastics needs 200 years in the soil (Bian Yousheng, 2005). With the unceasing expanding of mulching area of plastic film and the increasing of using years, the residual plastic film that was not degraded and continued to be accumulated in farmland, large numbers of residual film formed barrier layer gradually, which could hinder the development of crop root and the absorption of moisture and nutrients, hinder machinery tillage, damage crops growth environment, lead to soil compaction, poor permeability, reduction of crop yield and severe environmental pollution, the phenomenon that a large number of plastic film left in farmland make "white revolution" which has brought Gospel to agricultural production be transformed into "white pollution" (Liu Junke, 2000). In order to protect farmland ecological environment, domestic and international measures were actively developed and a variety of environmentally friendly biodegradable plastic film which can produce oxidized photochemical and biochemical effects were promoted, the use of biodegradable plastic film would be an effective way to completely solve the "white pollution" (Yang Huidi, 2000).

In recent years, biodegradable plastic film material and degradation process has become a research focus (Li Xianfa, 2004; Sun Jianping, et al, 2000; Zhang Chunhong et al, 2007). Currently, photolysis film, biodegradable film, photolysis/biodegradable film were researched more, the degradation process of film depended on biodegradation, photo-degradation and chemical degradation, and the effect of efficiency, synergy and coherence between the three main degradation process. Although a variety of biodegradable plastic film were developed in China, due to the high cost, the poor economy and the difficulty of

controlling the degradation of biodegradable film, the promotion and development were hindered. So, the development of degradable film which has good economy and low cost has become the main research direction.

In China, the construction of biogas started in the 1970s, and so far, with over 30 years of history. Biogas technology mainly used manure, straw and other organic substances as raw material to produce biogas by anaerobic fermentation (Zhang Yongmei, 2008). In recent years, the promotion and application of biogas technology was rapid in China, at the end of 2005, 17 million household biogas digesters and 140000 sewage purification digesters were built in rural areas, with an annual output of about 80 billions m³ gas. By the end of 2006, the total number of the rural household biogas has reached 22 millions, biogas construction had entered a new stage of rapid development. Currently, biogas residue were mainly used for planting, breeding, sideline and processing industry (Lu Mei et al, 2007; Guo Qiang, et al, 2005; RK Gupta, 2002), which was difficult to get high value resources utilization, so if its solid waste after anaerobic digestion could achieve high value resource utilization, the economic efficiency of industrial production of biogas could be improved and good ecological and social benefits would be produced (Tian Xin, 2008; Ye Xujun, et al, 2000).

In addition, because ruminant animals mainly fed on coarse fodder, such as rice straw, wheat straw, corn stalk, etc. The conversion rate of straw fibre was not high, the main reason for this phenomenon was that 20% to 70% of the fibre can not be degraded by rumen bacteria of ruminants, the result was that the biogas residue which was from ruminant manure fermentation contained large amounts of wood cellulose (An Juan, 2005).

Therefore, the full biodegradable biogas residue fibre film made of ruminant animal manure or fibre residue of straw fermentation for biogas and a certain percentage of plant fibre, using cleaner production processes, had moisture conservation and weeding property. The film could be completely degraded by microorganisms in the period of crop growth, restore in the soil, improve soil organic matter content, and meanwhile not pollute the environment, all of these would lay the foundation for applications and promotion of biodegradable film, to sustain agricultural sustainable development of our country had important realistic and historical significance.

1.1 Research status

1.1.1 Biogas residue utilization status

The research results showed that biogas residue was one of the residue after organic matter anaerobic fermentation for biogas, which was mainly composed of undecomposed raw materials solid and new generation microbial biomass (Lin Jianfeng, 2003). As cellulose, hemicellulose, lignin and other substance of fermentation materials remained in the biogas residue in the fermentation process, so biogas residue basically retains all the components of anaerobic fermentation production besides the gas. Biogas residue generally contains organic matter 36% to 49.9%, humic acid 10.1% to 24.6%, crude protein 5% to 9%, total nitrogen 0.8% to 1.5%, total phosphorus 0.4% to 0.6%, and total potassium 0.6% to 1.2%. The requirement of N,P,K of anaerobic fermentation process is very low, therefore, the majority of N,P,K and other elements of fecaluria were not being used, and eventually left in the biogas residue and biogas slurry (Bian Yousheng, 2005; Xie Tao, 2007). As microbial groups and undecomposed raw materials, so the biogas residue had its unique property (Zhang Quanguo, 2005).

Organic matter of biogas residue is not only a good fertilizer, but also conducive to microbial activity and the formation of granular structure, the organic matter surface can absorb amounts of soluble effective nutrients, under the soil microorganism's action, it can continuously provide compatible nutrients for growing (Zhang Wudi, 2003). Currently, the utilization of biogas residue is mainly as following (Guo Qiang et al., 2005):

1. Biogas residue fodder

Biogas residue contain 24 kinds of amino acid, many kinds of trace elements, B vitamins and other nutrients, biogas residue can be used to feed pigs, 50kg fodder can be saved and one month of fattening period can be shortened when fattening one pig. Feeding fish with biogas residue can not only improve fish yield and quality, but also reduce the occurrence of fish diseases, breeding earthworms with biogas residue can provide fodder of high-quality protein for livestock, while improve the utilization value of biogas residue.

2. Biogas residue fertilizer

Biogas residue is a high quality fertilizer, it can effectively improve soil physical and chemical property, increase soil organic matter and nutrient content, improve soil porosity, bulk density and water retention (Xu Shiwen, 1987).

3. Biogas residue adsorbent

The research which was made by C.Namasivayam (1995) showed that biogas residue can absorb heavy metal Cr^{6+} in wastewater better at pH was 1.5; biogas residue can absorb the "Direct red 12 B" stain in industry wastewater better at pH 2.7; biogas residue can absorb Pb^{2+} in wastewater better, and adsorption capacity can reach 28mg/g.

4. Biogas residue brewing

Using artificial culture and old kiln mud as bacteria, together with anaerobic biogas residue, the yield rate of wine increased 10.5% than without biogas residue (Lu Baoqing, 1997).

5. Biogas residue compost

Mixing biogas residue and straw with a 1 : 1 ratio was used for mushroom matrix. The biggest biogas industry was built in Nyirbator of Hungary (S.Ranik, 2004).

1.1.2 Biodegradable film research status

1.1.2.1 Research status of biodegradable film materials

Biodegradable materials research began in the 1960s. The initial study was mainly to add natural polymers with biodegradable properties (such as starch, etc.) to generic plastic, then get the so-called biodegradable materials. St.Lawrence starch-company developed a starch-polyethylene or polypropylene blends in Canada (Qiu Weiyang, 2002). With human understanding of biodegradability of macromolecule, the research focus began to turn to biodegradable materials (Qiao Haijun, 2007), which can be classified as microorganism synthetic polymer, chemical synthetic polymer and natural polymer.

Currently, the research of biodegradable film mainly focuses on the following aspects: (1) photo-degradation film, it is made of resin mixed with photo-sensitizer and accelerator. (2)

biodegradable film, it includes structural degradation film, biodegradable film containing inorganic salts and adding starch. (3) photo- biodegradable film; (4) plant fibre film.

In China, the major research was additive photo-degradation film and synthetic photo-degradation film. The research focused on using light stabilizers to control degradation period. Since 1997, 944-polymeric efficient light stabilizer, BW-6911 new light stabilizer were developed, which replaced the severe irritation and sensitization GW-504/2002 anti-aging system. American Dupont CO. , Ltd produced copolymers of ethylene and CO, American OCC and DOW CO. , Ltd had used this technology to produce film and develop industrial production (Xiong Hanguo, 2004). The disadvantages of photo-degradation film were susceptible to external environment, which was difficult to control the degradation period , and covering field, the part into soil can not be degraded, so its application was limited (Xu Xiangchun, 2006).

The degradation of biodegradable film was caused by microbes in natural environmental condition. It was divided into additive biodegradable film and completely biodegradable film according to degradation mechanism and damage style.

At present, additive biodegradable film was composed of plastic, starch, compatibility agents, self-oxidants, processing additives. Typical varieties were polyethylene starch biodegradable film (Liu Ming et al., 2008). There were institutes of physics and chemistry, Beijing University of Technology, Guangdong biodegradable plastic CO., Ltd more than 20 research institutes. The research focused adding starch or modified starch into PE.

The main varieties of completely biodegradable film were PLA, PCL, and PHB and so on. United States used PCL to produce synthetic polymer biodegradable film (KAM Abd I J-kader, 2002). Warner-Lambert developed a new type of resin, which was made of 70% amylopectin starch and 30% amylose starch (He Aijun, 2002). It had good biodegradability, was considered a significant development in material science.

Photo-degradation film was made of additive photo-sensitizer, auto-oxidants, and anti-oxidants as microbial culture medium in general polymer.

Plant fibre film has good ventilation, wet and dry strength and good biodegradability. Chinese academy of agricultural sciences successfully developed the environmentally-friendly hemp film (Fu Dengqiang, 2008). In addition, paper films composed of different materials were produced. South China Technology University used sugar cane and starch as materials to manufacture a kind of fully degradable film (Tan Chengrong, 2002). Japan manufactured biodegradable film with 1%-10% chitosan cloth softwood mechanical pulp original paper in 1990. The demand of environmental film increased in Washington State University, France, Germany, Italy, Canada, Netherlands and South Korea and other countries, leading to the environmentally-friendly film industry rapid development (Han Yongjun, 2008).

1.1.2.2 Research status of degradability of biodegradable film

In 1996, biodegradability of plant fibre paper was studied, which lower mechanical properties under certain environmental conditions, and eventually fragmented or completely degraded. Weight reduction and observation methods were employed by Gao Yujie (1996), Zhang Wenqun (1994), Wang Weigang (2003), Li Zhiming (2004) and Wang

Wei to study the biodegradability (2009), observation method only can describe the film degradation process and the weight reduction method can quantitatively explain the degradation process of biodegradable film.

In this chapter, the physical properties and chemical composition of the biogas residue produced by anaerobic fermentation using ruminant feces were determined and analyzed; manufacturing technology and performance of biogas residue film was studied by the methods of central composite quadratic rotatable orthogonal experiment; biogas residue fibre mulching for planting eggplant was studied with the method of random plot experiment.

2. The physical components of biogas residue and the chemical components of biogas residue fibre

2.1 The physical components of biogas residue

The determination of physical components of biogas residue was shown in table2-1.

Composition	Quality/ g	Percentages/%
Fibre	477	64
Non-mineral impurity	265	35
Mineral	8	1

Table 2-1. Biogas residue physical composition and their mass percentages

Table 2-1 showed that biogas residue was composed of three parts, fibre proportion was maximum, mineral proportion was minimum. There were mainly plastic, hair and grass seeds in the non-mineral impurities

2.1.1 Each component fibre and mineral content of biogas residue

The determination of each component fibre and mineral content was shown in table 2-2.

Group /mm	0.25~0.5	0.5~1	1~2	2~5	>5	total
Fibre quality /g	158	124	59	126	10	477
Minerals quality /g	0.8	5	1.3	0.7	0.2	8
Fibre quality percentages /%	21.10	16.50	7.90	16.80	1.40	64
Minerals quality percentages /%	0.11	0.67	0.17	0.09	0.03	1

Table 2-2. The fibre and minerals content and their mass percentages of each group

Table 2-2 showed that fibre quality percentage of 0.25 mm to 0.5 mm was maximum; fibre quality percentage of more than 0.5 mm was min; minerals quality percentage of 0.5 to 1 mm was maximum, minerals quality percentage of more than 5 mm was min.

2.1.2 Fibre morphology

Fig. 2-1 showed the fibre morphology of each group.

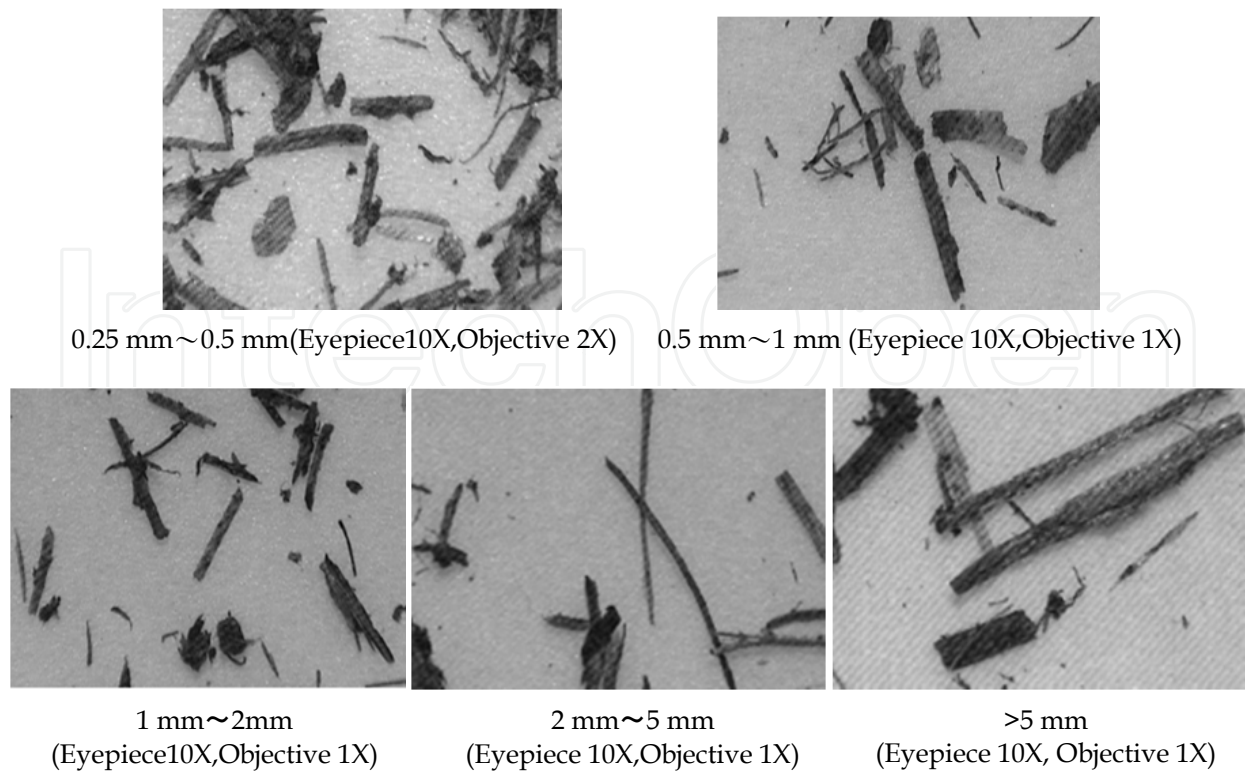


Fig. 2-1. Each component fibre morphology of the different amplification under microscope

2.1.3 The fibre length to width ratio

Determination results of the fibre length to width ratio of each group were measured and analysed by Motic Images Plus and shown in table 2-3.

Group /mm	Avg. L mm	L SD	L D range mm	Avg. W mm	W SD	W D range mm	Avg. L to W ratio	L to W ratio SD	L to W ratio D range
0.25~0.5	0.718	0.0990	0.52~0.92	0.060	0.0162	0.03~0.09	11.875	3.9790	3.92~19.83
0.5~1	1.940	0.1254	1.69~2.19	0.164	0.0432	0.08~0.25	11.845	1.6714	8.50~15.19
1~2	1.853	0.0873	1.68~2.03	0.142	0.0571	0.03~0.26	13.086	2.1457	8.79~17.38
2~5	2.493	0.3461	1.80~3.19	0.140	0.0345	0.07~0.21	17.726	4.2136	9.30~26.15
>5	3.491	0.6534	2.18~4.80	0.157	0.0318	0.09~0.22	22.174	4.9820	12.21~32.14

Notes: distribution estimates according to $\bar{x} \pm 2\sigma$, Avg. =average, L=length, W=width, D=distribution.

Table 2-3. Determination results of the fibre length to width ratio

Table 2-3 showed that average length distribution range was 0.52 mm to 4.80 mm, which of 0.25 mm to 0.5 mm was minimum, which of more than 5 mm was maximum, average width distribution range was 0.03 mm to 0.26 mm, which of 0.25 mm to 0.5 mm was maximum, which of 0.5 mm to 1 mm was maximum; average length to width ratio distribution range was 3.92 to 32.14, which of 0.5 mm to 1 mm was minimum, which of more than 5 mm was maximum. These showed that biogas residue length to width ratio had a greater dispersion. Although the average length to width ratio distribution range was smaller than straw fibre, which showed that it had a certain available value.

2.2 The chemical components of biogas residue fibre

Fig.2-2 showed the quality percentage of cellulose, hemi-cellulose, and lignin in biogas residue.

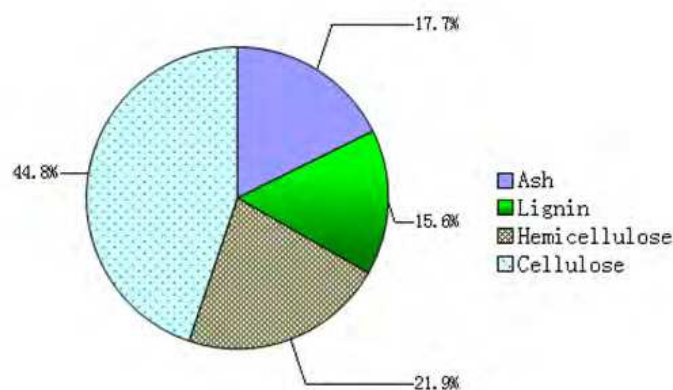


Fig. 2-2. Chemical compositions of biogas residue fibre

According to research results (Gao Zhenghua, 2008; Chen Hongzhang, 2008), the cellulose, hemi-cellulose, lignin of straw, wheat and corn stalks compared with these of biogas residue fibre were shown in table 2-4.

Species	Cellulose/%	Hemi-cellulose/%	Lignin/%	Ash/%
Straw	36.5	27.7	12.3	13.3
Wheat stalk	38.6	32.6	14.1	5.9
Corn stalk	38.5	28	15	4.2
Biogas residue fibre	44.8	21.9	15.6	17.7

Table 2-4. Chemical composition of biomass

The comparative analysis results showed that, cellulose quality percentage of biogas residue fibre after anaerobic fermentation was 5% higher than straw, wheat stalk, corn stalk; while hemi-cellulose quality percentage was 5% lower than straw, wheat stalk, corn stalk; lignin quality percentage did not change. The result showed that anaerobic fermentation to lignin content was not influence, hemi-cellulose relatively reduced, cellulose relatively increased, it is positive to the resources utilization of biogas residue.

3. The study on manufacturing technology of biogas residue film

Biogas residue fibre film samples were prepared with the method of clean pulping and paper-making process. The optimization of the technological parameters were studied by the method of the central composite quadratic orthogonal rotational experiment, beating degree, grammage, rosin, bauxite and wet strength agent were selected as input variables, and dry tensile strength, wet tensile strength, degradation period were chosen as response functions.

Factors and its levels of experiment were shown in table 3-1. Experimental plan and results were shown in table 3-2.

Factor level	Beating degree /SR°	Grammage /g/m ²	Rosin /%	Bauxite /%	Wet strength agent /%
Z _j	x ₁	x ₂	x ₃	x ₄	x ₅
γ(+2)	50	110	1.2	6	3.0
(+1)	45	95	1	5	2.4
(0)	40	80	0.8	4	1.8
(-1)	35	65	0.6	3	1.2
-γ(-2)	30	50	0.4	2	0.6

Table 3-1. Factors and its levels of experiment

Run	Factors				Response functions			
	Beating degree SR°	Grammage g/m ²	Rosin %	Bauxite %	Wet strength agent %	Dry tensile strength N	Wet tensile strength N	Degradation period day
	x ₁	x ₂	x ₃	x ₄	x ₅	y _{1i}	y _{2i}	y _{3i}
1	35	65	0.6	3	2.4	29.8	12.0	26.7
2	45	65	0.6	3	1.2	24.2	9.9	25.9
3	35	95	0.6	3	1.2	33.0	15.1	30.3
4	45	95	0.6	3	2.4	35.8	17.4	34.1
5	35	65	1	3	1.2	18.3	9.5	26.5
6	45	65	1	3	2.4	20.3	10.7	27.8
7	35	95	1	3	2.4	35.4	15.7	30.1
8	45	95	1	3	1.2	30.1	16.1	30.1
9	35	65	0.6	5	1.2	23.1	9.6	20.6
10	45	65	0.6	5	2.4	25.5	11.0	27.9
11	35	95	0.6	5	2.4	30.9	18.0	38.3
12	45	95	0.6	5	1.2	35.2	9.8	33.3
13	35	65	1	5	2.4	20.1	11.1	29.5
14	45	65	1	5	1.2	23.0	9.0	20.1
15	35	95	1	5	1.2	32.9	13.0	32.1
16	45	95	1	5	2.4	35.5	17.0	38.9
17	30	80	0.8	4	1.8	26.3	14.7	30.5
18	50	80	0.8	4	1.8	23.4	12.0	31.1
19	40	50	0.8	4	1.8	18.0	9.7	25.0

Run	Factors				Response functions			
	Beating degree SR°	Grammage g/m ²	Rosin %	Bauxite %	Wet strength agent %	Dry tensile strength N	Wet tensile strength N	Degradation period day
	x ₁	x ₂	x ₃	x ₄	x ₅	y _{1i}	y _{2i}	y _{3i}
20	40	110	0.8	4	1.8	40.5	19.5	37.0
21	40	80	0.4	4	1.8	33.8	16.4	29.4
22	40	80	1.2	4	1.8	26.4	12.6	30.6
23	40	80	0.8	2	1.8	25.9	11.4	31.1
24	40	80	0.8	6	1.8	26.9	11.1	32.5
25	40	80	0.8	4	0.6	22.1	8.7	26.4
26	40	80	0.8	4	3	26.6	15.5	37.2
27	40	80	0.8	4	1.8	28.0	13.4	32.8
28	40	80	0.8	4	1.8	32.3	13.3	33.4
29	40	80	0.8	4	1.8	29.6	12.7	33.2
30	40	80	0.8	4	1.8	30.6	13.8	31.6
31	40	80	0.8	4	1.8	28.8	11.8	33.6
32	40	80	0.8	4	1.8	27.6	12.3	36.8
33	40	80	0.8	4	1.8	31.1	14.5	36.7
34	40	80	0.8	4	1.8	33.2	14.9	35.1
35	40	80	0.8	4	1.8	31.8	14.4	34.6
36	40	80	0.8	4	1.8	30.7	14.7	41.1

Table 3-2. Experimental plan and results

3.1 Degradation period test

The arrangement of the degradation period test was shown in Fig.3-1. Degradation state of film samples during degradation period was shown in Fig.3-2



Fig. 3-1. The arrangement of the degradation period test

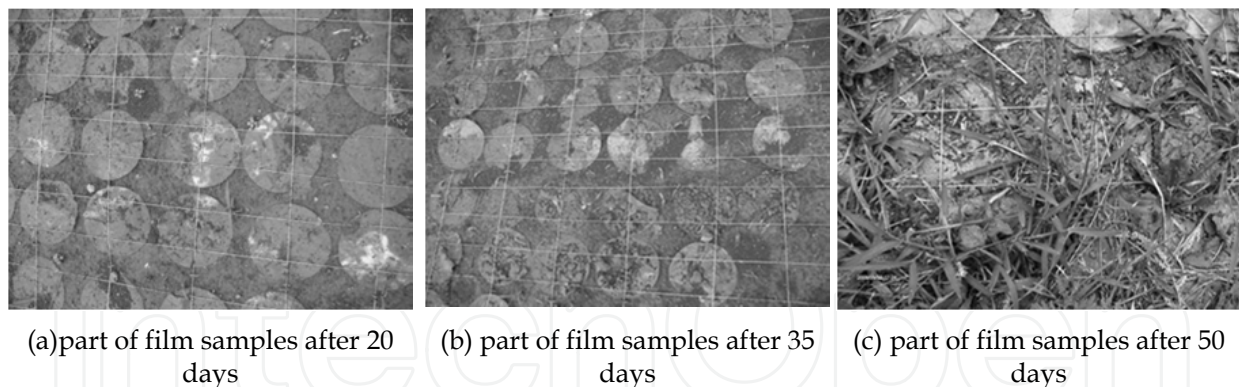


Fig. 3-2. Mulching degradable process in the different time during the period of degradation test

The fibre film of biogas residue for degradation discovered that the appearance and performance of the samples changed a lot because of light, air temperature, air humidity, wind, rain and other weather factors, coupled with the soil temperature, humidity, combined effect of microorganisms. According to the observation, the degradation was divided into several stages, initially, the sample surface appeared holes or small cracks, called induction period of the film degradation; over time, holes and cracks gradually expanded, the edge glued to the soil surface, the role of soil microorganisms on the film samples increased, resulted in an increasing number of small holes, broken into fragmentation period of the film samples, the film samples effected by various types of micro-organisms would become increasingly thin, the mechanical strength decreased gradually, until the film entered into the fast degradation period of the samples. Especially, after rain, the increasing of air humidity and soil humidity would make mechanical strength of the samples decrease rapidly, so soil moisture is an important impact factor of the film degradation.

During degradation of the film, the dry tensile strengths were regularly measured, according to scatter, the trends of dry tensile strength (N) and date (d) were available, according to trend line, the time of dry tensile strength at zero of each group was estimated, which was defined degradation period. The result was shown in table 3-2.

3.2 Response model

3.2.1 Response model

Response models of dry tensile strength, wet tensile strength and degradation period at $\alpha=0.05$, were significant and the models were shown as equation 3-1, 3-2 and 3-3.

$$y_1=30.38+9.135 \times 10^{-3}x_1+5.4x_2-1.52x_3+0.067x_4+0.94x_5-1.02x_1^2-0.64x_4^2-1.15x_5^2+1.14x_1x_4+1.24x_2x_3+0.96x_3x_4-1.11x_4x_5 \quad (3-1)$$

$$y_2=13.81-0.36x_1+2.47x_2-0.35x_3-0.36x_4+1.43x_5-0.62x_4^2-0.42x_5^2+0.64x_1x_3+0.46x_2x_5-0.44x_3x_5+0.65x_4x_5 \quad (3-2)$$

$$y_3=34.95+0.22x_1+3.59x_2+0.017x_3+0.5x_4+2.33x_5-1.12x_1^2-1.07x_2^2-1.32x_3^2-0.87x_4^2-0.87x_5^2+1.67x_2x_4+1.41x_4x_5 \quad (3-3)$$

3.2.2 Analysis of importance of various factors on response functions

Importance of various factors on response functions was shown in table 3-3.

Source	Importance		
	Dry tensile strength (N)	Wet tensile strength (N)	Degradation period (D)
Beating degree	1.645	1.046	0.905
Grammage	1.838	1.323	2.341
Rosin	1.094	1.333	0.931
Bauxite	1.344	1.951	2.099
Wet strength agent	1.526	2.839	2.253

Table 3-3. Importance of each factor

3.3 Effect of interaction factors on dry tensile strength

3.3.1 Effect of beating degree and bauxite on dry tensile strength

Fig.3-3 showed the effect of beating degree and bauxite on dry tensile strength when other factors were held at 0 level. With the increase of beating degree and bauxite, dry tensile strength firstly increased and then slowly decreased, the maximum occurred when the two factors were held at 0 level. The reason was that with the beating degree increased, the fibre sub-wire broom degree was high, the exposure of hydrogen bonding of the fibre surface increased, the bonding forces between the fibres enhanced, so that the film strength increased; when beating degree was more than a certain value, the single fibre strength was destroyed, the bonding force decreased, led to the decrease of strength, adding bauxite excessively to strength had side effect, thus resulting in strength decreased.

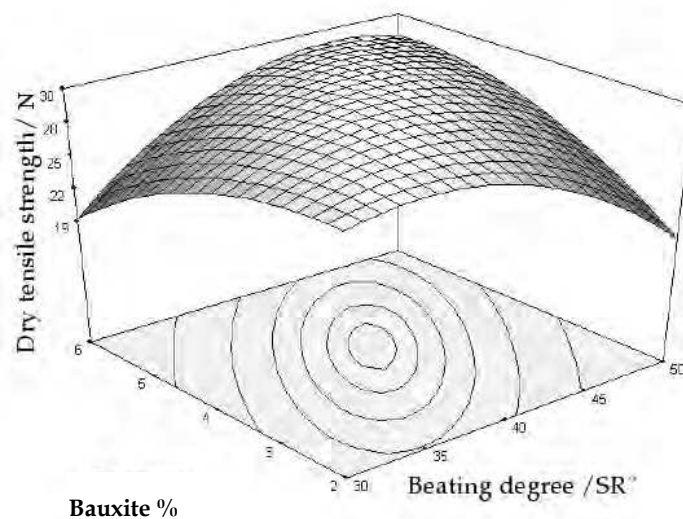


Fig. 3-3. Response surface and contour plots for the effects of beating degree and bauxite on dry tensile strength: grammage was held at 80 g/m² and rosin was held at 0.8%, wet strength agent was held at 1.8%

3.3.2 Effect of grammage and rosin on dry tensile strength

Fig.3-4 showed the effect of grammage and rosin on dry tensile strength when other factors were held at 0 level. Dry tensile strength significantly increased with the increase of grammage; when grammage was small, dry tensile strength slowly decreased with the amount of rosin increases, this was because bonding effect of the additive rosin to fibre became larger, when the grammage was large, dry tensile strength slowly increased with the amount of rosin increase, the maximum occurred when rosin was held at 1.2%, and grammage was held at 110 g/m². With the grammage increased, the number of fibre in per area increased, bonding between the fibres enhanced, the strength increased, at this moment, the positive effect of grammage on strength was much greater than the negative impact of rosin.

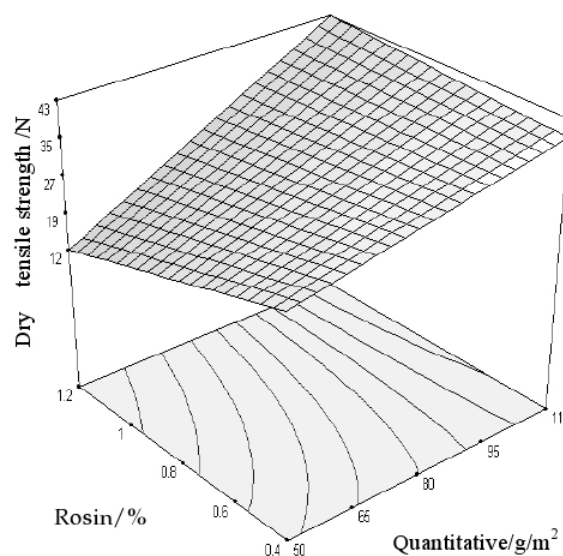


Fig. 3-4. Response surface and contour plots for the effects of grammage and rosin on dry tensile strength: beating degree was held at 40SR°, bauxite was held at 4%, wet strength agent was held at 1.8%

3.3.3 Effect of rosin and bauxite on dry tensile strength

Fig.3-5 showed the effect of grammage and rosin on dry tensile strength when other factors were held at 0 level. Adding the amount of bauxite at a low level, the dry tensile strength decreased with the increase of added rosin amount; adding the amount of bauxite at a high level, the added rosin amount almost had no effect on the dry tensile strength, maximum of the dry tensile strength occurred when bauxite was held at 4%, and rosin was held at 0.4%, because rosin adsorption has been saturated, there was no effect on the strength.

3.3.4 Effect of bauxite and wet strength agent on dry tensile strength

Fig.3-6 showed the effect of bauxite and wet strength agent on dry tensile strength when other factors held at 0 level. When bauxite was near 0 level, the dry tensile strength increased with the increase of wet strength agent, when bauxite was higher than the zero level, with the wet strength agent increased, the dry tensile strength first increased and then

decreased, the maximum occurred when wet strength agent was held at 2%, and bauxite was held at 3.5%. This is because with the adding of the wet strength agent, the adsorption of the fibre system to wet strength agent had already been saturated, and it no longer played a role in increasing strength, anionic trash in absorption system impacted the combination between the fibre, leading to strength decreased.

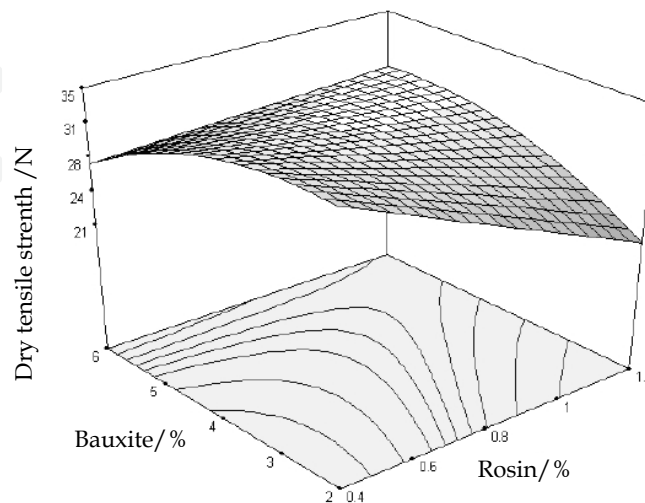


Fig. 3-5. Response surface and contour plots for the effects of rosin and bauxite on dry tensile strength: beating degree was held at 40SR°, grammage was held at 80 g/m², wet strength agent was held at 1.8%

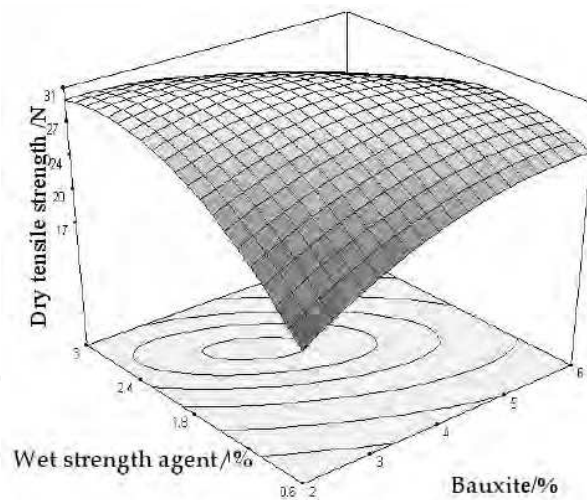


Fig. 3-6. Response surface and contour plots for the effects of bauxite and wet strength agent on dry tensile strength: beating degree was held at 40SR°, grammage was held at 80 g/m², rosin was held at 0.8%

3.4 Effect of interaction factors on wet tensile strength

3.4.1 Effect of beating degree and rosin on wet tensile strength

Fig.3-7 showed the effect of beating degree and rosin on wet tensile strength when other factors were held at 0 level. When the beating degree was lower than 0 level, wet tensile

strength decreased with the increase of rosin; when the beating degree was higher than 0 level, wet tensile strength increased with the increase of beating degree and rosin, the maximum value occurred when beating degree was held at 30SR°, and rosin was held at 0.4%. This is because added rosin impacted adsorption effect of fibre to wet strength agents, wet tensile strength decreased, but with the continuing increase of beating degree, the fibre sub-wire broom degree further enhanced, the adsorption effect of fibre on wet strength agent was over than the rosin.

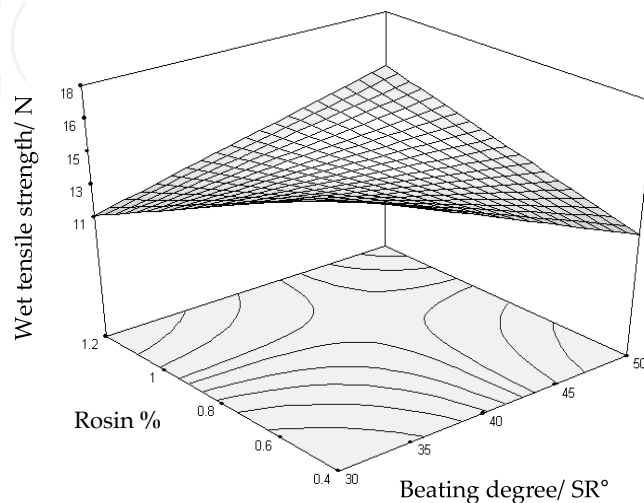


Fig. 3-7. Response surface and contour plots for the effects of beating degree and rosin on wet tensile strength: grammage was held at 80 g/m², bauxite was held at 4%, wet strength agent was held at 1.8%

3.4.2 Effect of grammage and wet strength agent on wet tensile strength

Fig.3-8 showed the effect of grammage and wet strength agent on wet tensile strength when other factors were held at 0 level. Wet tensile strength gradually increased with the increase

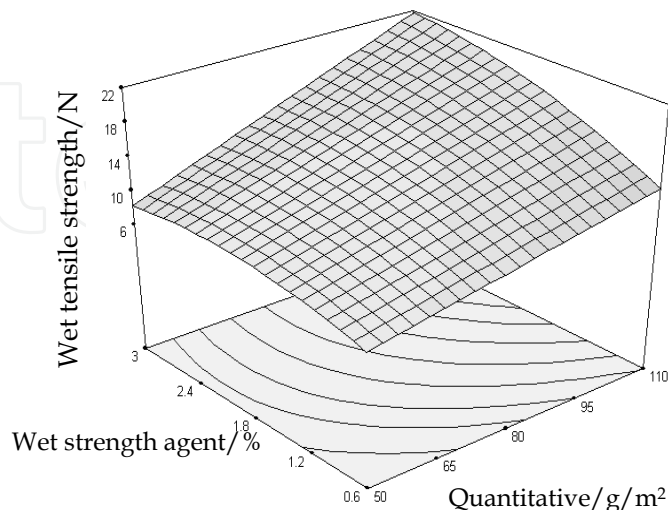


Fig. 3-8. Response surface and contour plots for the effects of grammage and wet strength agent on wet tensile strength: beating degree was held at 40SR°, rosin was held at 0.8%, bauxite was held at 4%

of the grammage and wet strength agent; the maximum occurred when wet strength agent was held at 3%, and grammage was held at 110 g/m², this is because the number of fibre increased and bonding effect of fibre enhanced, when wet tensile strength increased with the increase of grammage, at the same time, wet strength agent provided cationic charge, fibre strongly adsorbed wet strength agent added because of pulp fibre with anionic charge, so that the wet tensile strength of film increased.

3.4.3 Effect of rosin and wet strength agent on wet tensile strength

Fig.3-9 showed the effect of rosin and wet strength agent on wet tensile strength when other factors were held at 0 level. When rosin was lower than 0 level, wet tensile strength increased with the increase of wet strength agent; When rosin was higher than 0 level, the increase of wet tensile strength became flat with wet strength agent increased, the maximum occurred when wet strength agent was held at 3%, and rosin was held at 0.4%. The reason was that the increase of rosin, affected the adsorption of the fibre to wet strength agent, to a certain extent, reduced the effect of wet strength agent, wet tensile strength would not increase or decrease.

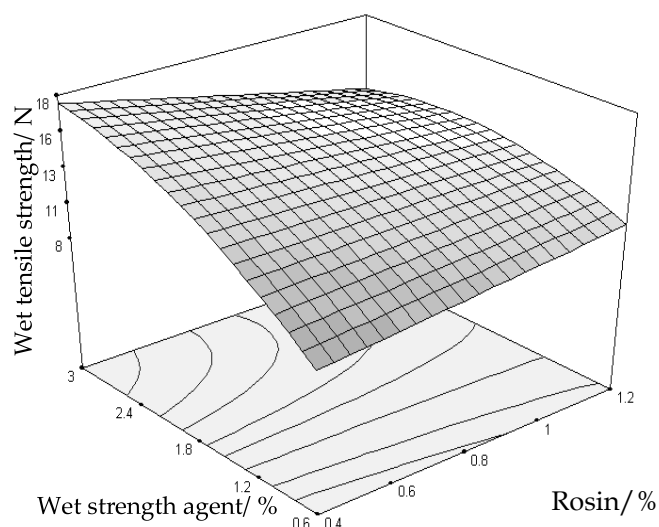


Fig. 3-9. Response surface and contour plots for the effects of rosin and wet strength agent on wet tensile strength: beating degree was held at 40SR°, grammage was held at 80 g/m², bauxite was held at 4%

3.4.4 Effect of bauxite and wet strength agent on wet tensile strength

Fig.3-10 showed the effect of bauxite and wet strength agent on wet tensile strength when other factors were held at 0 level. When bauxite was at any level, wet tensile strength gradually increased with the increase of wet strength agent ;When bauxite was lower than 0 level, wet tensile strength increased, When bauxite was higher than 0 level, wet tensile strength decreased, the maximum occurred when wet strength agent was held at 3%, and bauxite was held at 4.5%.The reason was that the increased amount of added bauxite in the slurry system, led to adsorption of anionic trash in fibre system, affected the adsorption to the wet strength agent , resulted in decrease of wet tensile strength.

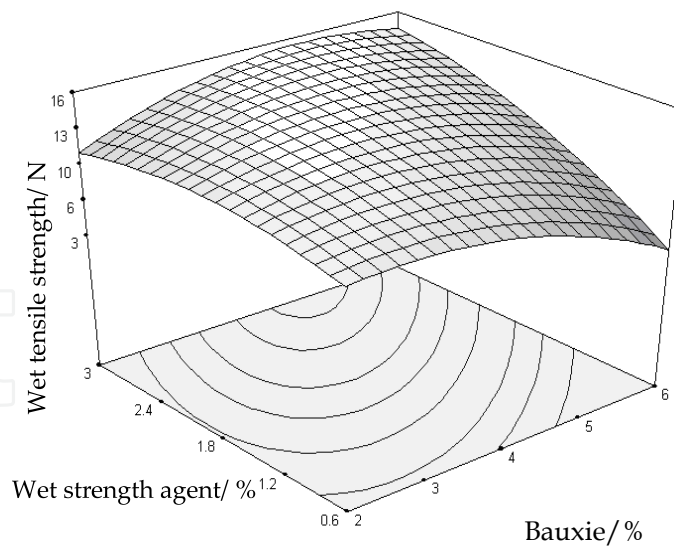


Fig. 3-10. Response surface and contour plots for the effects of bauxite and wet strength agent on wet tensile strength: beating degree was held at 40SR°, grammage was held at 80 g/m², rosin was held at 0.8%

3.5 Effect of interaction factors on degradation period

3.5.1 Effect of grammage and bauxite on degradation period

Fig.3-11 showed the effects of grammage and bauxite on degradation period when other factors were held at 0 level. Degradation period gradually increased with the increase of bauxite and grammage, the maximum occurred when bauxite was held at 6%, and grammage was held at 110 g/m², because of the increase of grammage, the number of fibres grew, the bonding capacity between fibres enhanced, the amount of bauxite increased, the ability of fibre that absorbing additives enhanced, which made dry tensile strength and wet tensile strength of film become larger, the degradation period of film increase.

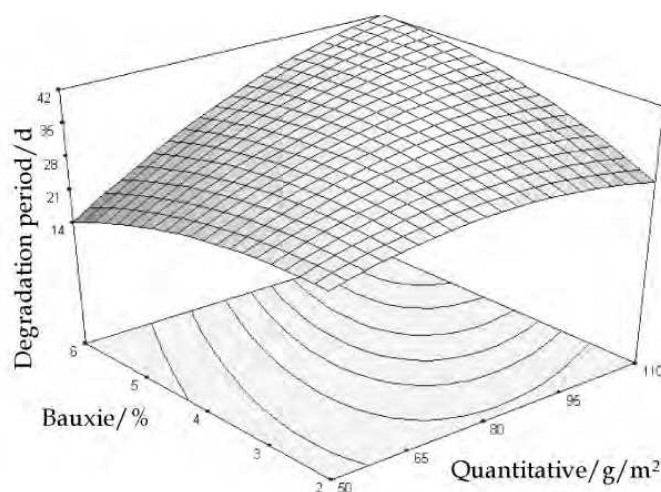


Fig. 3-11. Response surface and contour plots for the effects of grammage and bauxite on the degradation period: beating degree was held at 40SR°, rosin was held at 0.8%, wet strength agent was held at 1.8%

3.5.2 Effect of bauxite and wet strength agent on degradation period

Fig.3-12 showed the effects of bauxite and wet strength agent on degradation period when other factors were held at 0 level. Degradation period gradually increased with the increase of bauxite and wet strength agent, the maximum occurred when bauxite was held at 6%, and wet strength agent was held at 3%, wet strength agent at a suitable amount could improve the wet tensile strength of film, and make degradation period grow.

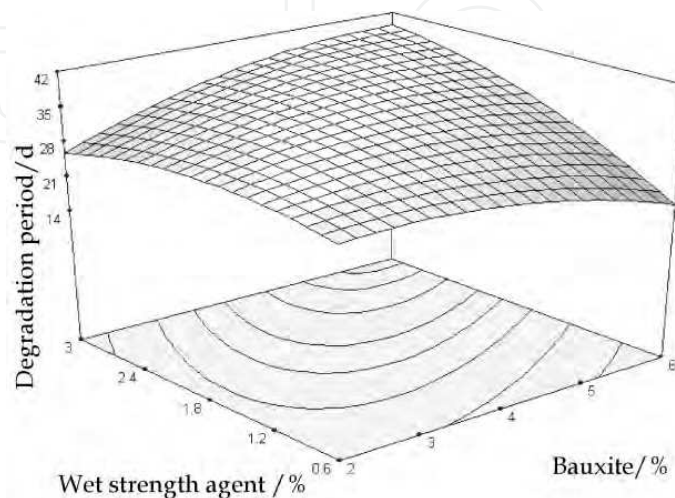


Fig. 3-12. Response surface and contour plots for the effects of bauxite and wet strength agent on the degradation period: beating degree was held at 40SR°, grammage was held at 80 g/m²,rosin was held at 0.8%

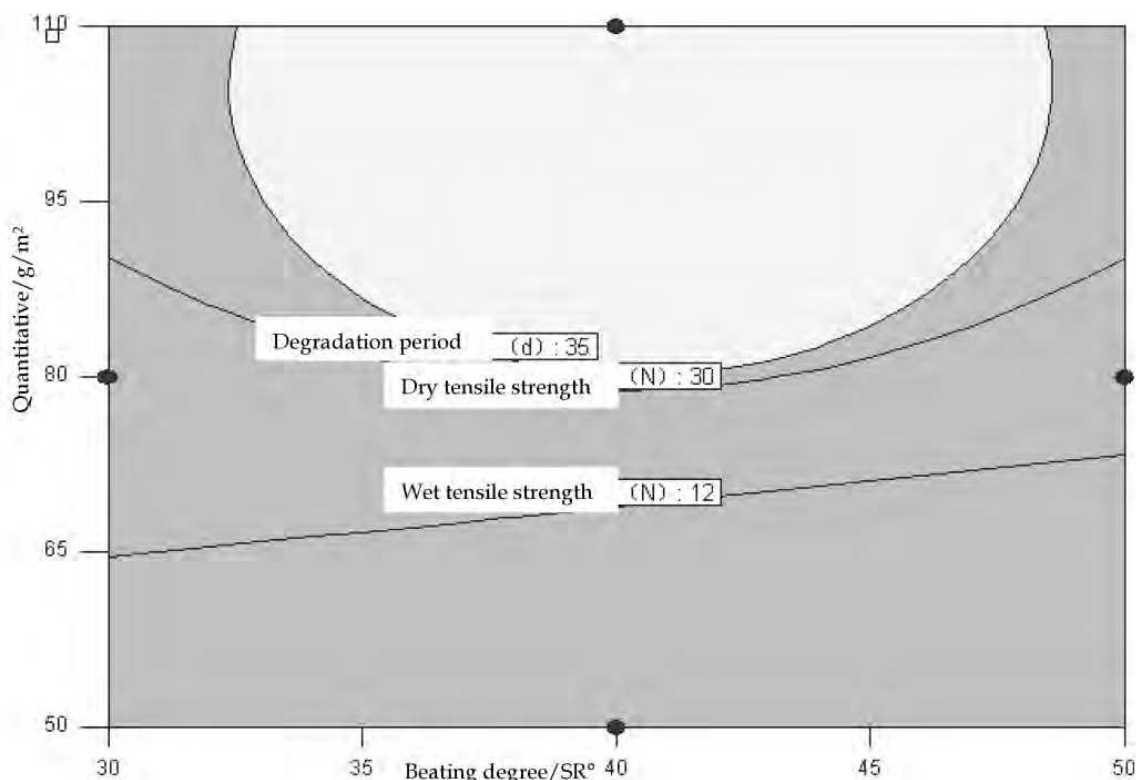


Fig. 3-13. Optimum analysis plot: rosin was held at 0.8%, bauxite was held at 4%, wet strength agent was held at 1.8%.

3.6 Optimization

The rule of optimization based on film performance meeting the agronomic requirement to reduce energy consumption and save raw materials as much as possible was applied to determine the optimum combination of the factors. The result was that when rosin was held at 0.8%, bauxite was held at 4%, wet strength agent was held at 1.8%, beating degree was held at 35 SR°, grammage was held at 80 g/m², the performance that was dry tensile strength was greater than 30N, wet tensile strength was greater than 12N, the degradation period was 35 days to 60 days could be obtained, seeing in Fig 3-13.

3.7 Manufacturing technology

Based on the above research results, manufacturing technology of the biogas residue fibre film was obtained, seeing Fig 3-14.

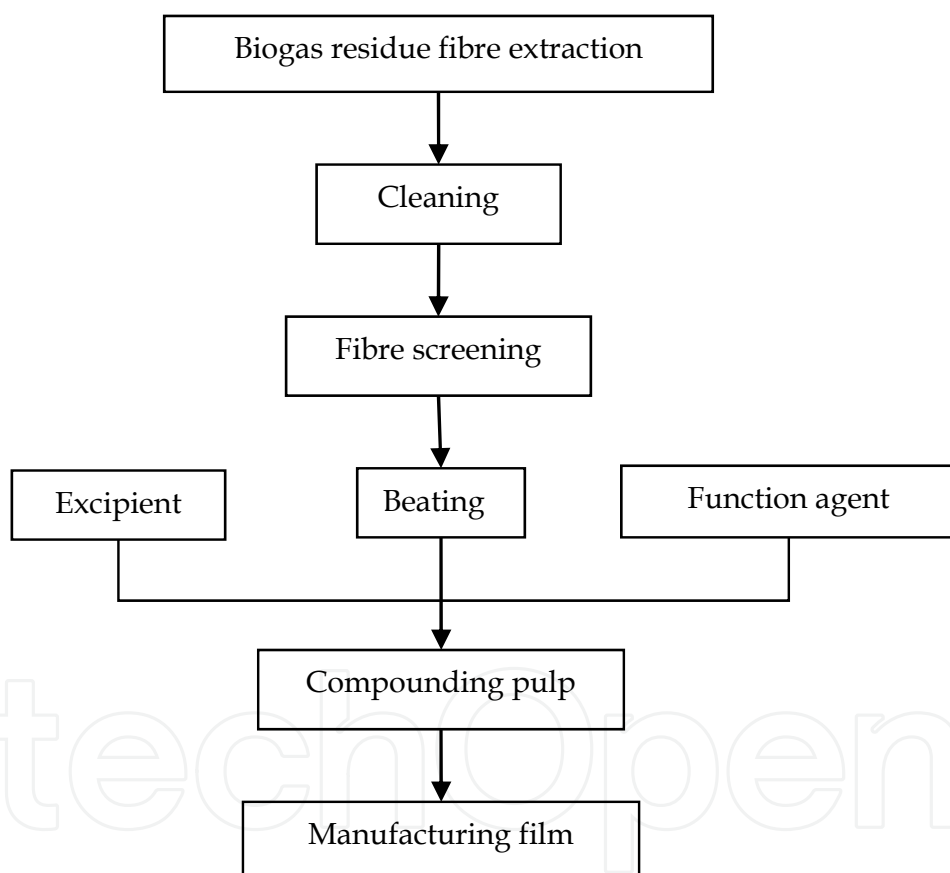


Fig. 3-14. Manufacturing technology of the biogas residue fibre film

4. The experiment of cultivating eggplants with biogas residue fibre film

In order to test the performance differences among the three kinds of biogas residue fibre mulch, black biodegradable mulching added in activated carbon, plastic film and control (without mulching), to cultivate eggplant, a comparative field test through the multiple comparisons for the soil moisture content, soil temperature, weed growth amount and eggplant yield was employed. Performance index of the treatments was shown in table4- 1.

Treatment	Grammage g/m ²	Dry tensile strength N	Wet tensile strength N	Thickness mm	Width cm
A	65	33.70	12.46	0.248	70
B	80	36.30	14.40	0.316	70
C	100	30.69	10.40	0.385	70
D	55	39.16	13.46	0.099	70
E	9	1.50	-	0.011	70

Notes: A,B,C were biogas residue fibre mulch of different grammage, respectively; D was black biodegradable mulching added in activated carbon; E was plastic film.

Table 4-1. Performance index of the treatments

4.1 Effect of different treatments on the weed amount

The effect scene of different treatments on the weed amount in the case of cultivating eggplant was shown in Fig.4-1. Weed amount of mulching was markedly less than the control, and weed amount of plastic film was more than biogas residue films. In the period of observation, weeds were flourished under the plastic film or even broke plastic mulch. Weeds mainly grew from transplanted hole and rupture to all the treatments, and making the film rupture expands.

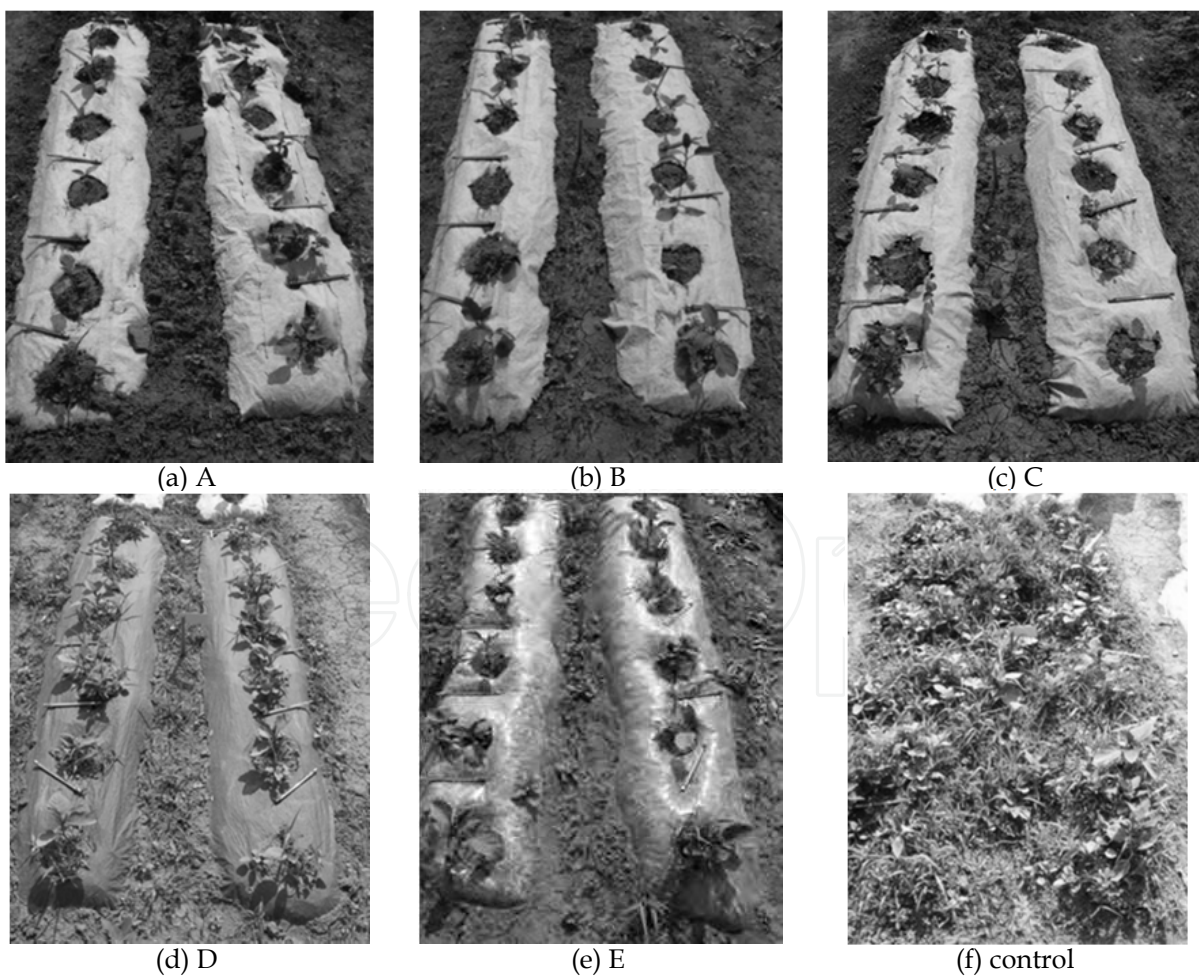


Fig. 4-1. The effect scene of different treatments on the weed amount in the case of cultivating eggplant

The measured results of total weeds of each treatment were shown in table 4-2.

Interval\Treatment	Weed amount (g)					
	A	B	C	D	E	F
1	63.01	79.02	48.15	69.19	430.7	528
2	90.02	66.88	79.26	72.6	204.99	376.05
3	88.96	79.06	79.62	87.38	328.85	358.63
Mean	80.66	74.99	69.01	76.39	321.51	420.89

Table 4-2. The measured results of total weeds of each treatment

Analysis of variance of the weed amount was shown in table 4- 3.

Source	SS	DF	MS	F	P-value	F _{0.05}
Interval	9089.895	2	4544.947	1.290108	0.317365	4.1028
Treatment	365347.3	5	73069.46	20.74117	5.51E-05	3.3258
Error	35229.19	10	3522.919			
Total	409666.4	17				

Table 4-3. Analysis of variance of the weed amount

There were significant differences among the treatments, according to analysis of the multiple comparisons among treatments, seeing table 4-4.

Treatment	Mean difference (g)						LSD 0.05	LSD 0.01
	\bar{y}_i	$ \bar{y}_i - \bar{y}_F $	$ \bar{y}_i - \bar{y}_E $	$ \bar{y}_i - \bar{y}_D $	$ \bar{y}_i - \bar{y}_C $	$ \bar{y}_i - \bar{y}_B $		
A	80.66	340.23**	240.85**	4.27	11.65	5.67	108	154
B	74.99	345.9**	246.52**	1.4	5.98			
C	69.01	351.88**	252.5**	7.38				
D	76.39	344.5**	245.12**					
E	321.51	99.38						
F	420.89							

Notes: \bar{y}_i was mean of the i treatment; LSD_{0.05} and LSD_{0.01} was significant at 0.05, not significant at 0.01.

Table 4-4. Multiple comparisons of the weed amount among treatments

The results showed that there were significant differences between three kinds of biogas residue fibre film, black film and plastic film, control; there were significant differences between three kinds of biogas residue fibre films and black film. Weed amount of A biogas residue fibre film decreased 81% as compared with control, and decreased 75% as compared with the plastic film; weed amount of B biogas residue fibre film decreased 82% as

compared with control, and decreased 77% as compared with the plastic film; weed growth amount of C biogas residue fibre film decreased 84% as compared with control, and decreased 79% as compared with the plastic film. From this, three kinds of residue fibre films had significant effect of suppressing weeds.

4.2 Effect of different treatments on soil moisture

The soil moisture content of each treatment and analysis of variance of the soil moisture content were respectively shown in table 4-5 and table 4-6.

Interval\Treatments	A	B	C	D	E	F
1	19.49	19.29	19.31	18.06	20.97	17.82
2	20.53	20.33	19.35	18.25	19.43	15.4
3	19.21	18.52	20.15	18.15	21.04	16.47
Mean	19.74	19.38	19.61	18.16	20.48	16.56

Table 4-5. The soil moisture content of each treatment

Source	SS	DF	MS	F	P-value	F _{0.05}
Interval	0.2636	2	0.1318	0.1777	0.8398	4.1028
Treatments	29.7137	5	5.9427	8.0105	0.0028	3.3258
Error	7.4187	10	0.7419			
Total	37.3960	17				

Table 4-6. Analysis of variance of the soil moisture content

Table 4-6 showed that there were significant differences among treatments. Multiple comparisons of the soil moisture content among treatments were shown in table 4-7.

Treatment	Mean difference (%)						LSD _{0.05}	LSD _{0.01}
	\bar{y}_i	$ \bar{y}_i - \bar{y}_T $	$ \bar{y}_i - \bar{y}_T $	$ \bar{y}_i - \bar{y}_T $	$ \bar{y}_i - \bar{y}_C $	$ \bar{y}_i - \bar{y}_C $		
A	19.74	3.18**	0.74	1.58*	0.13	0.36	1.56	2.23
B	19.38	2.82**	1.1	1.22	0.23			
C	19.61	3.05**	0.87	1.45*				
D	18.16	1.6*	2.32**					
E	20.48	3.92**						
F	16.56							

Notes: The significance of symbols was as same as table 4-4.

Table 4-7. Multiple comparisons of the soil moisture content among treatments

The results showed that there were significant differences between three kinds of biogas residue fibre film, plastic film and control; there were significant differences between black film and control; there were significant differences between plastic film and black film. There were significant differences between A, C of biogas residue fibre film and black film;

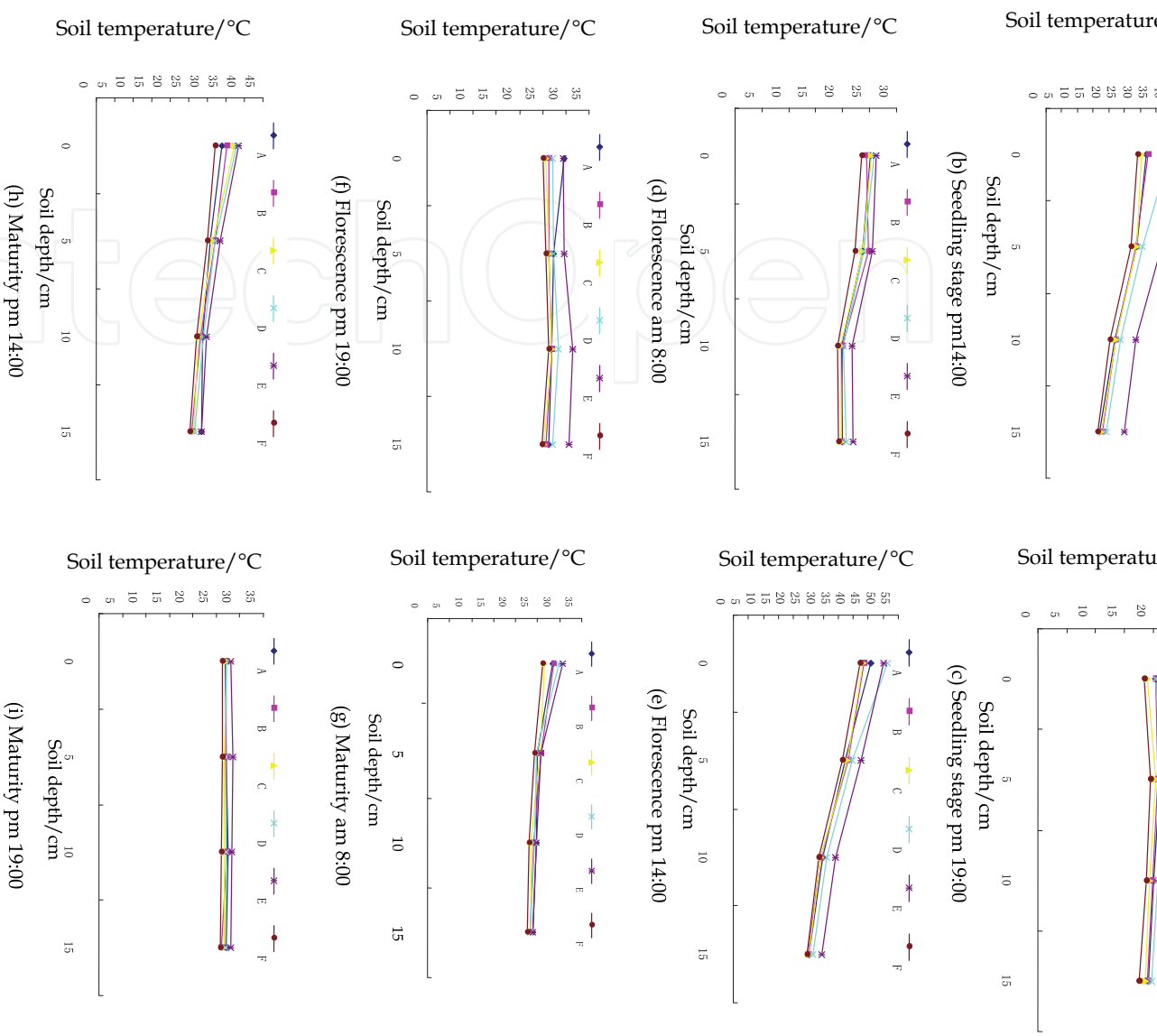


Fig. 4-2. The soil temperature that varied with soil depth of the six treatments

there were not significant differences between B of biogas residue fibre film and black film; there were not significant differences between three kinds of biogas residue fibre film and plastic film. It can be seen, soil moisture of biogas residue fibre film was significantly higher than control and black film, and there were not significant difference as compared with plastic film.

4.3 The effect of different treatments on soil temperature

4.3.1 The effect of soil depth on soil temperature among treatments in different growth stages

The soil temperatures that varied with soil depth of the six treatments were shown in Fig.4-2.

Figure 4-2 showed that the mulching temperature was higher than the control during the whole growth period, the temperature of plastic film was the highest, the temperature of black film was higher than the biogas residue fibre film, and the temperature of A, B, C of the biogas residue fibre film was slightly higher than the control. It could be seen from the temperature curve of am7:00 and pm 14:00, soil temperature gradually decreased with the soil deepening, but, the temperature of soil surface decreased at pm 19:00, the soil temperature slightly increased with soil deepening in the stage of revival and flowering, and the soil temperature was constant in maturity; mulching had a certain warming effect in the stage of revival and flowering, and had not warming effect in maturity, the reason was that the film had been degraded.

4.3.2 Effect of different treatments on total accumulated temperature

The measured results of the total accumulated temperature of each treatment were shown in table 4-8.

Soil depth cm	Interval\ treatments	A	B	C	D	E	F
0	1	435.6	438.4	427.5	463.2	526.9	396.1
	2	408.7	403.9	415.8	430	479.6	401.8
	3	418.1	410.2	369.6	431.3	511.8	369.1
	Mean	420.8	417.5	404.3	441.5	506.1	389
5	1	395	380.5	395.6	410	435.7	380.8
	2	393.1	397.2	386.8	392.4	425.4	377.6
	3	380.7	397.4	391.8	405.7	446.5	370.5
	Mean	389.6	391.7	391.4	402.7	440.1	374.7
10	1	356.5	356	343.2	366.4	381.3	350.4
	2	365.8	367.1	358.5	378.2	407.2	335.7
	3	362.8	354.8	359.4	367.2	413	344.4
	Mean	361.7	359.3	353.7	370.6	400.5	343.5
15	1	339.2	332.4	330.5	346.7	370.4	337.1
	2	348.1	346	346.6	359.8	385.5	342.3
	3	342.9	341.3	335.4	348.6	398.8	323.8
	Mean	343.4	339.9	337.5	351.7	384.9	334.4

Table 4-8. The total accumulated temperature of each treatment

Analysis of variance of the total accumulated temperature of each treatment was shown in table 4-9.

Soil depth cm	Source	SS	DF	MS	F	P-value	F _{0.05}
0	Interval	3016.57	2	1508.29	6.3092	0.0169	4.1028
	Treatment	25517.2	5	5103.44	21.3477	4.84E-05	3.3258
	Error	2390.63	10	239.06			
	Total	30924.4	17				
5	Interval	58.83	2	29.42	0.3999	0.6806	4.1028
	Treatment	6241.93	5	1248.39	16.9726	0.000133	3.3258
	Error	735.53	10	73.55			
	Total	7036.3	17				
10	Interval	324.96	2	162.48	2.1843	0.1633	4.1028
	Treatment	5774.55	5	1154.91	15.526	0.000195	3.3258
	Error	743.86	10	74.39			
	Total	6843.37	17				
15	Interval	432.25	2	216.13	4.113	0.0497	4.1028
	Treatment	5264.62	5	1052.92	20.037	6.42E-05	3.3258
	Error	525.47	10	52.55			
	Total	6222.34	17				

Table 4-9. Analysis of variance of the total accumulated temperature of each treatment

Multiple comparisons of the total accumulated temperature of different soil depths among treatments were shown in table 4-10.

Table 4-10 showed that there were significant differences between plastic film and three kinds of biogas fibre residue film, black film and control while the soil depth was 0cm and 10cm. There were significant differences between the black film and control; there were significant differences between A, B treatment of biogas residue fibre film and control; there were no significant differences between C treatment of biogas residue fibre mulch and control; there were significant differences between black film and C treatment of biogas residue fibre film. While the soil depth was 5 cm, there were significant differences between plastic film and three kinds of biogas fibre residue film, black film and control; there were significant differences between A, B treatment of biogas fibre residue film, black film and control. While soil depth was 15 cm, there were significant differences between plastic film and three kinds of biogas fibre residue film, black film and control; there were significant differences between the black film and C treatment of biogas residue fibre film and control.

While the soil depth was 0 cm, 5 cm, 10 cm, the total accumulated temperature of A, B treatment of biogas residue film obviously increased than control, and C did not significantly increase; while the soil depth was 0cm, the total accumulated temperature of A treatment increased 31.8 °C, B increased 28.5 °C, and C increased 15.3°C than control; while the soil depth was 5cm, the total accumulated temperature of A treatment increased 17°C, B increased 17.3°C, and C increased 15.2°C than control; while the soil depth was 10cm, the total accumulated temperature of A treatment increased 18.2 °C, B increased 15.8 °C, and C increased 10.2 °C than control. While the soil depth was 15 cm, the total accumulated temperature of three kinds of biogas fibre residue film nearly closed to control, and was less than the black film and plastic film. There was no significant difference between the three kinds of biogas residue fibre film.

Soil depth (cm)	Treat ment	Mean difference (°C)						LSD _{0.05}	LSD _{0.01}
		\bar{y}_i	$ \bar{y}_i - \bar{y}_F $	$ \bar{y}_i - \bar{y}_E $	$ \bar{y}_i - \bar{y}_D $	$ \bar{y}_i - \bar{y}_C $	$ \bar{y}_i - \bar{y}_B $		
0	A	420.8	31.8*	85.3**	20.7	16.5	3.3	28.13	40.01
	B	417.5	28.5*	88.6**	24	13.2			
	C	404.3	15.3	101.8**	37.2*				
	D	441.5	52.5**	64.6**					
	E	506.1	117.1**						
	F	389							
5	A	391.4	17*	48.7**	11.3	1.8	0.3	15.6	22.19
	B	391.7	17.3*	48.4**	11	2.1			
	C	389.6	15.2	50.5**	13.1				
	D	402.7	28.3*	37.4**					
	E	440.1	65.7**						
	F	374.4							
10	A	361.7	18.2*	38.8**	8.9	8	2.4	15.68	22.32
	B	359.3	15.8*	41.2**	11.3	5.6			
	C	353.7	10.2	46.8**	16.9*				
	D	370.6	27.1**	27.1**					
	E	400.5	57**						
	F	343.5							
15	A	343.4	9	41.5**	8.3	5.9	3.5	13.19	18.76
	B	339.9	5.5	45**	11.8	2.4			
	C	337.5	3.1	47.4**	14.2*				
	D	351.7	17.3*	33.2**					
	E	384.9	50.5**						
	F	334.4							

Notes: The significance of symbols was as same as table 4-4.

Table 4-10. Multiple comparisons of the total accumulated temperature of different soil depths among treatments

4.4 The effect of different treatments on eggplant yield

Analysis of variance of the yield was shown in table 4-11.

Source	SS	DF	MS	F	P-value	F _{0.05}
Interval	0.5030	2	0.2515	2.4037	0.1405	4.1028
Treatment	3.7059	5	0.7412	7.0833	0.0045	3.3258
Error	1.04637	10	0.1046			
Total	5.2553	17				

Table 4-11. Analysis of variance of the yield date

Table 4-11 showed that there were significant differences between the yields of each treatment. Multiple comparisons of the eggplant yield among treatments were shown in table 4-12.

Treatment	Mean difference (kg)						LSD _{0.05}	LSD _{0.01}
	\bar{y}_i	$ \bar{y}_i - \bar{y}_F $	$ \bar{y}_i - \bar{y}_E $	$ \bar{y}_i - \bar{y}_D $	$ \bar{y}_i - \bar{y}_C $	$ \bar{y}_i - \bar{y}_B $		
A	1.95	0.42	1.00**	0.50	0.07	0.30	0.58	0.82
B	2.25	0.72*	0.70*	0.20	0.37			
C	1.88	0.35	1.07**	0.57				
D	2.45	0.92**	0.50					
E	2.95	1.42**						
F	1.53							

Notes: The significance of symbols were as same as table4-4.

Table 4-12. Multiple comparisons of the eggplant yield among treatments

The results showed that there were significant differences between B treatment of biogas residue fibre film and control, no significant differences between A, C treatment and control, significant differences between black film and control. There were extremely significant differences between the plastic film, A, C treatment and control, there were significant differences between plastic film and B treatment, no significant differences between plastic film and the black film. It can be seen, the yield of B treatment increased 47% as compared with control.

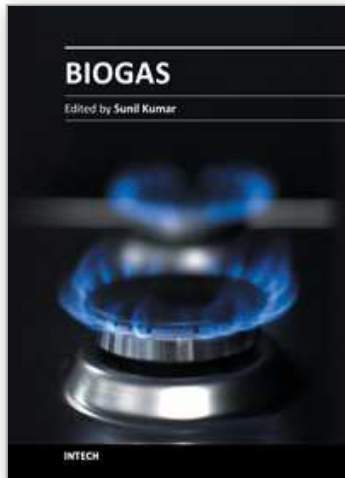
5. Conclusions

1. Biogas residue are mainly composed of the fiber, non-metallic minerals, minerals, their mass percentage are 64%, 35% and 1% respectively; cellulose, hemicellulose, lignin and ash are 44.8%,21.9%,15.6% and 17.7% respectively in the biogas residue fiber. The mass percent of biogas residue cellulose is over 5% more than that of rice straw, wheat straw and corn stalk; the mass percent of the hemicellulose is 5% less than that of rice straw, wheat straw and corn stalk.
2. An optimum factors combination is rosin 0.4%, bauxite 4% and wet tensile strength 1.8%, beating degree 40SR°, grammage 80 g/m², in this case, for the biogas residue fibre film, dry tensile strength can attain more than 30N , wet tensile strength can attain more than12N, the degradation period can attain 35 days to 60 days.
3. The rank of importance of the five factors on the dry tensile strength: grammage, beating degree, wet strength agent, bauxite and rosin; on the wet tensile strength: wet strength agent, bauxite, rosin, grammage and beating degree; on the degradation: grammage, wet strength agent, bauxite, rosin and beating degree.
4. Biogas residue fibre film has significant effect against weeds as compared with plastic film and bare field.
5. There was no significant difference of conserving moisture between biogas residue fiber mulching and the plastic film.
6. In the period of eggplant growth, the rank of the total accumulated temperature: plastic film, black film, biogas residue fibre film and bare field.
7. The eggplant yield of biogas residue fiber mulching whose grammage is 80g/m² is 47% more than bare field, and slightly lower than that of the plastic film.
8. It is feasible to make biodegradable mulching from the biogas residue, in accordance with the agronomic requirement

6. References

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