



## CHAPTER IV

### RESULTS AND DISCUSSIONS

#### Results of Degummed Waste Silk

Shinano Kenshi (Thailand) Co., Ltd. reported the result of degumming waste silk (facsimile transmission, February 2, 1994) as follows :

Mass before degumming	18.6 kg
Dry mass after degumming	9.64 kg
Degumming loss	51.83 %

According to degumming loss of waste silk, it was found that waste silk from Keba had sericin more than silk filaments. The degummed waste silk was whiter in color, softer and more lustrous. Unfortunately the degummed silk mass held together tightly, and it was difficult to loosen the mass by hand. Consequently, it can not be used in the available carding machine in this thesis. A opener may be used to open up the lumps of degummed silk and screen the trash, but this machine was not available in this thesis.

### **Results of Web Formation**

During the carding process, some waste silk fell out from the carding machine, caused the web having areal density less than the required value. In order to compensate the loss weight, waste silk was weighed more than the determined weight in Table 3.1 about 10 to 20 g. The falling out of waste silk may be because silk fibers are too fine to card with the roller carding machine and waste silk had a slight amount of crimp. A crimp is necessary for formation of a manageable web for nonwoven production (Buresh, 1962).

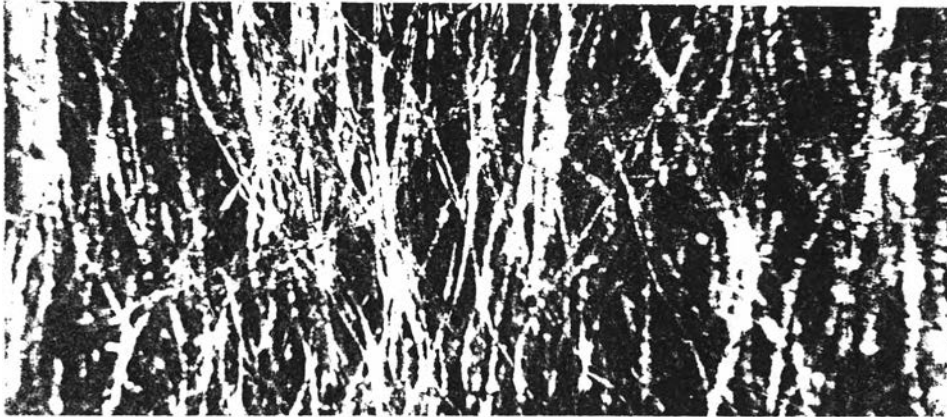
### **Appearance of Nonwoven Fabrics**

The appearance of waste silk nonwoven fabrics to the naked eyes was yellowish white and lustrous. They also had a soft hand and was noticed that the fibers lied roughly parallel to one another with some black spots and trash scattering over the fabrics. Some areas of the fabrics were observed that the bonding points were attached together; as a result those areas were smooth, stiff and paperlike. This may be caused by the ununiform distribution of binder fibers. The nonwoven used polyolefin binder fibers can be seen this bonding area clearly. This can be explained by the fact that polyolefin flowed more easily than polyester at the same temperature. However the time and temperature used in thermalbonding process were also critical to the characteristic of bonding points. Krcma (Newton and Ford, 1973) has discussed the formation of a point structure by means of core-sheath bicomponent fibers that the bonding of fibers should be at the point of contact only, rather than over large areas of binder material. He has stressed the strict control of

conditions, which is needed for the optimum structure to be obtained. In regard to softness 60PET20 was quite softer than 60PET35 distinctly, and 60PET25 and 60PET30 was in the middle. This was agreed to the information and trends reported by Moncriefe (1975) and Hoyle (1990) confirming that more binder contents resulted in more stiffness and harsh handle.

### **Microscopic Examination**

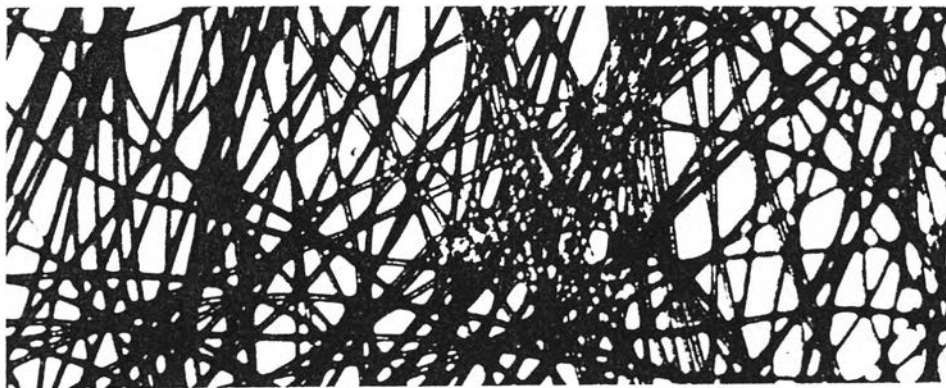
The fiber arrangement and the appearance of bonding point were reproduced by the light microscope and the scanning electron microscopes. Figure 4.1 showed more detail of fiber arrangement of silk nonwoven fabric compared to Sontara and Skawol. It was apparent that the silk nonwoven produced by a carding machine was the parallel type fabric which fibers has parallelized orientation where Sontara and Skawol had a random arrangement. It was also found in Figure 4.1 (a) that waste silk fibers were translucent, and highly lustrous with slightly crimped.



(a) The silk nonwoven fabric taken at 9 magnification



(b) Sontara taken at 20.625 magnification



(c) Skawol taken at 20.625 magnification

Figure 4.1 Optical photomicrographs of the silk nonwoven fabrics compared to the available nonwoven products in the market.

The enlarged view in Figure 4.2 showed the location of bonding points of silk nonwoven fabric taken by the Hitachi scanning electron microscope. It was noted that the bonding of fibers provide a network structure. As mentioned above, waste silk fibers were slightly crimped. In consequence, the network of bonding points in the fabrics was not complex. Marcher (1991) have observed the strength of the reinforcing network and it was found that a crimped fiber with a zig-zag structure gave a somewhat higher number of contact points, and increasing the binder fiber content can provide more bonding points.

Figure 4.3 showed the characteristic of a bonding point of the silk nonwoven fabric. The thermoplastic fibers used as binder were combined several fibers around them when they melt. By using the JEOL scanning electron microscope, the sample must have sputtered with gold to conduct electrons, so the sheath and core of bicomponent fibers can not be observed. In order to study the bonding mechanism of bicomponent binder fibers, the Olympus light microscope was again used, and then the bonding points were taken, and shown in Figure 4.4. Thermal bonding by such bicomponent fibers was accomplished by melting the sheath component when its melting point was obtained, followed by adhesion of the molten sheath at contact points between the fibers nearby. After cooling, the core component which have a high melting point compared to the sheath component, provide a strong supporting network. In Figure 4.4, the sheath and core components of binder fibers were clearly observed.



┌ 50 microns

Figure 4.2 The scanning electron photomicrograph showing the location and the network structure of bonding points of the silk nonwoven fabric by Hitachi scanning electron microscope.



┌ 100 microns

Figure 4.3 The enlarged view of the bonding point taken by JEOL scanning electron microscope.

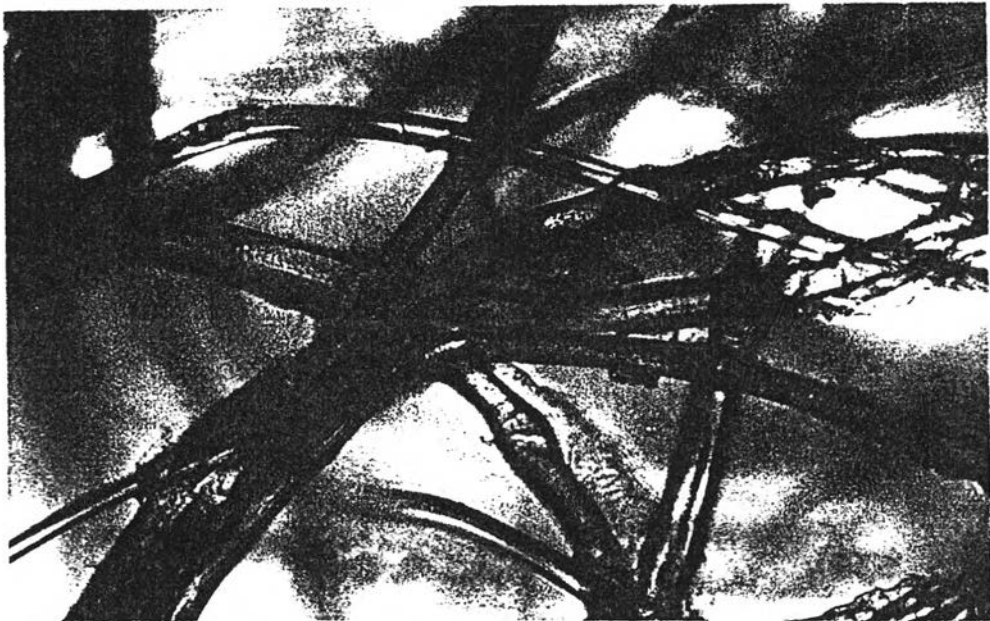


Figure 4.4 The optical photomicrograph showing the bonding mechanism of bicomponent fibers taken at 82.5 magnification.

### Areal Density and Uniformity

Table 4.1 summarised the average values of thickness and actual areal density of the silk nonwoven fabric samples and the products available in the market. It was found that the actual areal densities of all samples were somewhat different from the prepared areal densities. The reason for this revealed in the blending stage. The waste silk was excessively weighed to compensate the loss fibers during the carding. In addition to the uneven spreading of the fiber mass into the feed-in rollers, the agglomeration of fibers in some parts of the web may occur. Skawol had the areal density about  $20 \text{ g/m}^2$ , two times less than Sontara, while silk nonwoven products had the actual areal density about  $50\text{-}70 \text{ g/m}^2$ .

The coefficients of variation of thickness which were a measure of the uniformity of the nonwoven fabrics (Cusick et al., 1963) fell into the acceptable range of 8-10% in comparison with the range 5% and 10% of Sontara and Skawol respectively.



Table 4.1 Average thickness and actual areal density of the silk nonwoven fabrics.

Samples	Thickness, mm			Actual Areal Density, g/m <sup>2</sup>		
	AVE.	S.D.	%C.V.	AVE.	S.D.	%C.V.
SONTARA <sup>(1)</sup>	0.248	0.013	5.09	43.06	2.28	5.28
SKAWOL <sup>(2)</sup>	0.172	0.012	9.17	20.39	1.13	5.56
4OPE30	0.401	0.027	6.79	59.48	3.23	5.43
6OPE30	0.484	0.025	5.08	82.50	10.14	12.29
4OPET30	0.285	0.013	4.42	51.58	3.38	6.55
6OPET20	0.378	0.036	9.62	61.67	5.01	8.12
6OPET25	0.345	0.028	8.04	64.52	7.81	12.10
6OPET30	0.421	0.044	10.38	72.97	5.66	7.75
6OPET35	0.327	0.030	9.15	65.89	5.78	8.77

(1), (2) : The available nonwoven products in the market (Atsawahem and Wiraset, 1993).

### Moisture Regain

The moisture regain is expressed as the amount of water resorbed by a dried fabric at equilibrium with standard condition (JIS L 1096, 1990). The average moisture regain of silk nonwoven fabrics can be found in Table 4.2. The range were 2.0 - 3.6%. In general, silk is noted for its high moisture regain (11%) while the moisture regain of the synthetic fibers such as polyester and polyolefin is so much lower than that of any of the natural fibers as neglect to 0.4%. It was reported by Wongwiboonporn et al. (1992) that the 100% waste silk nonwoven had the moisture percentage about 5.6%. This could be because waste silk had sericin more than the silk filaments and when they was thermal bonded, some fibers were covered by the molten binder fibers. Therefore, the synthetic binder fibers increased, the moisture regain should have decreased. But the results from Table 4.2 disagreed with this assumption. This must have been because the accurated waste silk and binder fiber weight were not prepared, and the actual contents were not analysed.

Table 4.2 Average moisture regain of the silk nonwoven fabrics.

Samples	Moisture regain, %
40PE30	2.02
60PE30	3.62
40PET30	2.87
60PET20	3.19
60PET25	3.66
60PET30	3.02
60PET35	2.68

### Air Permeability

The samples prepared were tested for the air permeability. The average air permeability of five samples were obtained and shown in Table 4.3. It was apparent that the products from waste silk had the air permeability more than  $160 \text{ cm}^3 \text{ s}^{-1} \text{ cm}^{-2}$ . In comparison with the cotton nonwoven fabric and the waste silk propylene blanket described in Table 2.1 and Table 2.2 respectively, silk nonwoven fabrics showed a better air permeability. They were also better than the general woven fabrics which the average air permeability were not more than  $100 \text{ cm}^3 \text{ s}^{-1} \text{ cm}^{-2}$ . However, it was expected that increasing binder fiber contents will lead to decreasing in air permeability the more bonding points will obstruct air flow (Ozsanlav, 1994).

Table 4.3 Average air permeability of some silk  
nonwoven fabrics.

Samples	Air Permeability, $\text{cm}^3 \text{s}^{-1} \text{cm}^{-2}$
40PE30	198.2
60PE30	164.6
40PET30	192.6
60PET20	194.6
60PET30	168.6

### **Tensile Properties**

The average maximum stress, elongation at break and initial modulus of silk nonwoven fabrics in both machine and cross-machine directions were evaluated for tensile strength. Figure 4.5 showed the typical stress-strain curve of the silk nonwoven fabrics which correlation between the maximum load and elongation was plotted. All specimens in machine direction behaved in the same way like Figure 4.5. When a specimen was loaded with the constant rate, it elongated or straightened relating to the load applied, until the fabric broke.

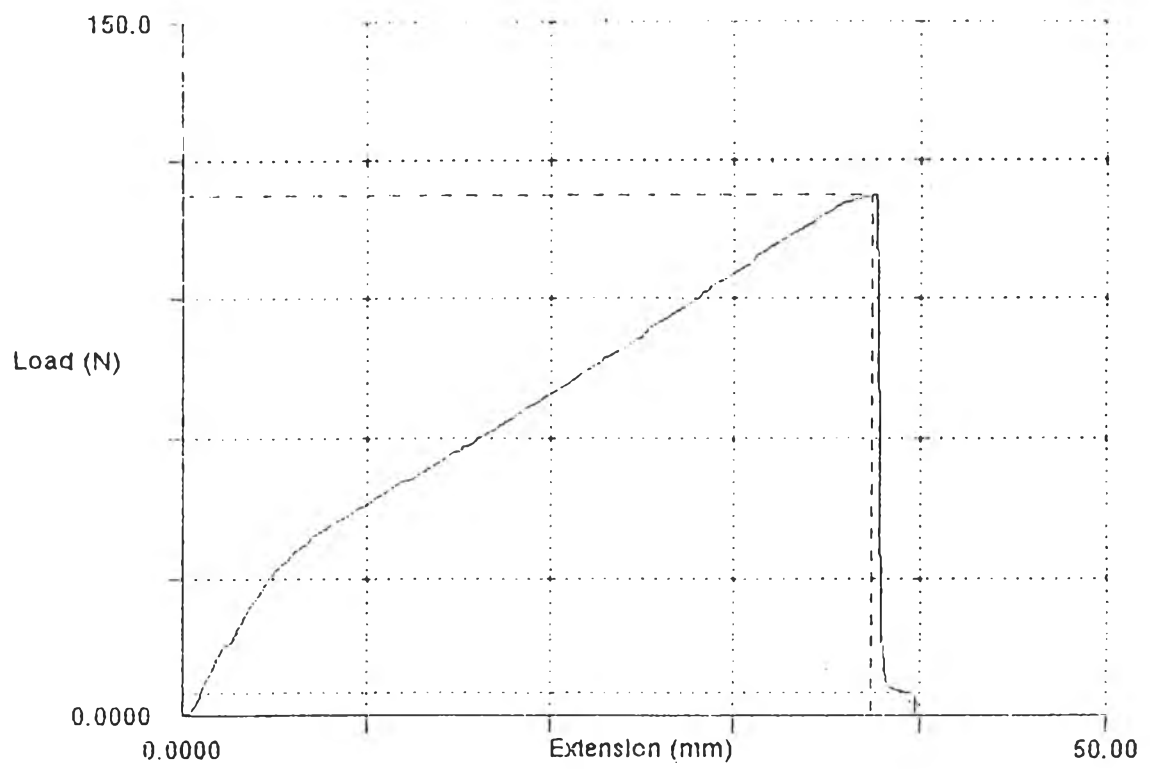


Figure 4.5 Typical stress-strain curve of the silk nonwoven fabrics.

## 1. Tensile Strength

The average maximum stress with coefficients of variation was found in Table 4.4. No other prepared samples except 60PET30 and 60PET35 provided the average maximum stress which could be comparable to Sontara and Skawol in both machine and cross-machine directions. It was noted that the maximum stress in cross-machine direction of all samples were considerably less than that in machine direction. This could be obviously seen in Figure 4.6, when the bar chart of the maximum stress of each sample was drawn. This confirmed the fact that the nonwoven fabrics produces from carding machine had high machine-directioned strength but rather low cross-machine directioned strength (Bernard, 1985). The maximum stress in cross-machine direction of silk nonwoven fabrics were ten times less than in machine direction while Sontara and Skawol which fibers arranged randomly, were only 3-4 times in difference.

Gibson and McGill (1987) studied the failure mechanism of thermal bondable polyester fibers, explained that there were essentially three ways in which a calender-bonded nonwoven can fail when stretched. First, adhesive failure can occur between the fibers within the bond site. Second, individual fibers can fracture either near the attachment point to the bond site or somewhere in the fiber between two bond sites. Third, the bond site itself can fracture. The failure almostly occured in the first way, but bonding temperature increased, the method of failure tended to change form that of first mechanism to primarily the second.



Table 4.4 Average tensile strength of the silk nonwoven fabrics in the machine and cross-machine directions.

Samples	Maximum Stress in Machine Direction, N/m <sup>2</sup>			Maximum Stress in Cross-machine Direction, N/m <sup>2</sup>		
	AVE.	S.D.	%C.V.	AVE.	S.D.	%C.V.
SONTARA <sup>(1)</sup>	10.66	0.67	6.29	3.94	0.29	7.24
SKAWOL <sup>(2)</sup>	11.18	0.91	8.15	2.83	0.35	12.32
40PE30	7.93	1.28	16.20	0.42	0.11	26.25
60PE30	5.66	1.76	31.18	0.32	0.10	29.34
40PET30	7.13	1.47	20.60	0.67	0.20	30.02
60PET20	4.26	0.55	12.94	0.27	0.05	19.99
60PET25	7.21	1.11	15.43	0.31	0.03	9.77
60PET30	10.21	1.41	13.83	0.90	0.06	6.20
60PET35	13.69	0.94	6.87	0.61	0.07	11.17

(1), (2) : The available nonwoven products in the market (Atsawahem and Wiraset, 1993).

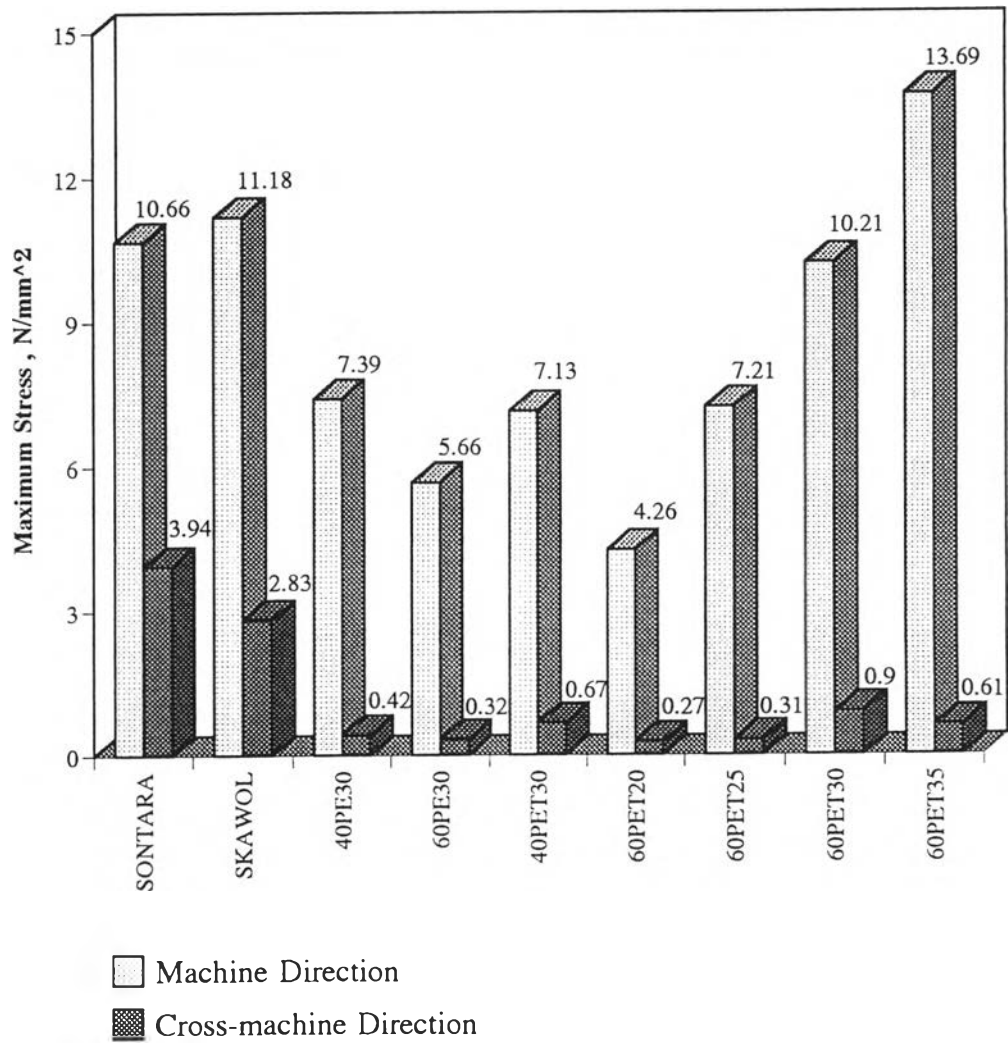


Figure 4.6 Comparison between the maximum stress in machine and cross-machine directions of silk nonwoven fabrics.

In Table 4.4, it was also observed that increasing the binder fiber contents gave the increasing in maximum stress. The plot of the PET binder fiber contents and maximum stress in machine was shown in Figure 4.7. This trend agreed with the works of Bernard (1985), Hoyle (1990), Marcher (1991), and Ozsanlav (1994). the observations of percentages which were varied, were only four. Thus the forward trend when more than 35% of binder fibers can not be predicted.

Marcher (1991) also discussed that a high strength can be obtained even at low binder fiber content if the sufficient network structure was formed. Increasing the content further will only give moderate increases in strength, since the only change will be in network density. Ozsanlav (1994) studied in the calender-bonded jute, found that an increase from 15% to 30% synthetic content in all cases almost trebled the fabric strength and extensibility.

However the coefficients of variation of the maximum stress were rather high values of 10-30% , showing the great ununiformity of the nonwoven fabrics produced compared to the available products which the variation was about 10%.

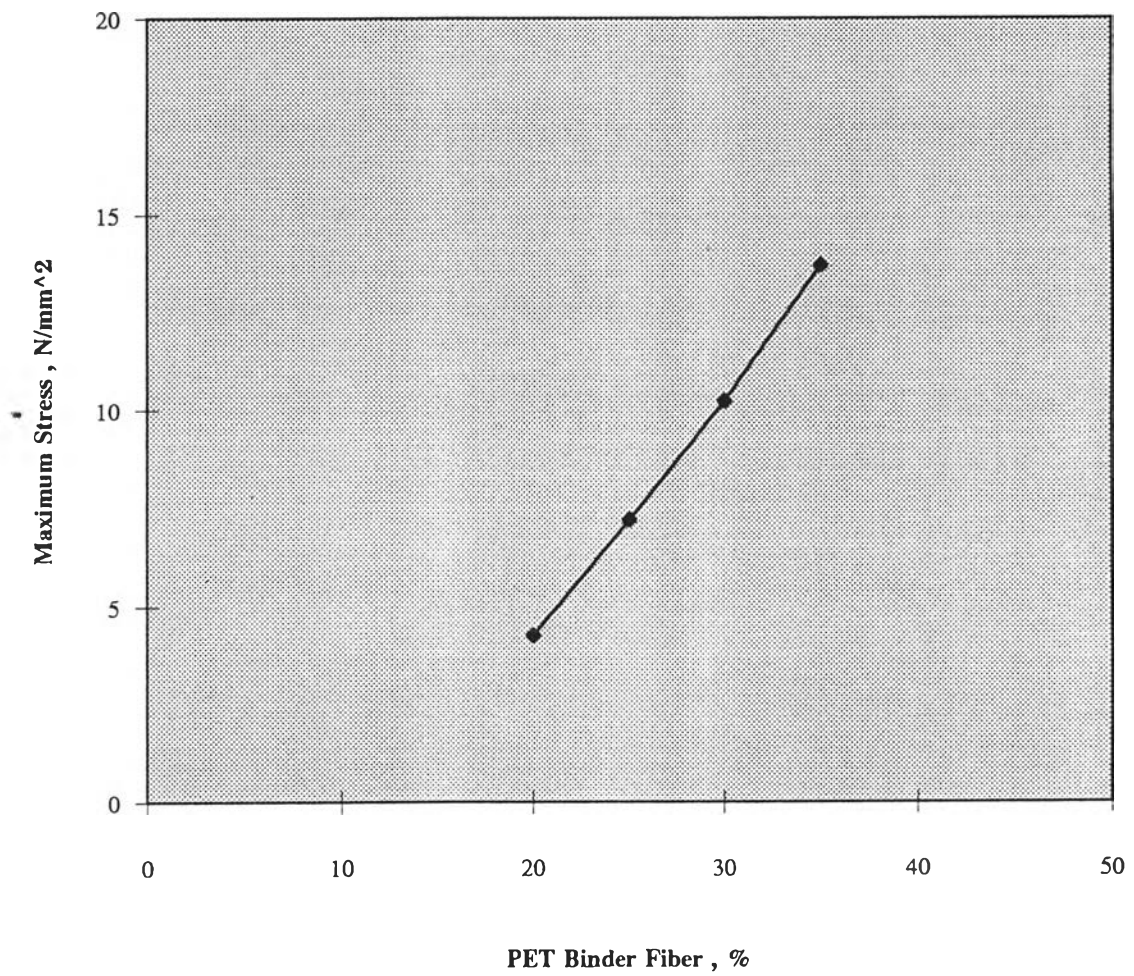


Figure 4.7 Correlation between PET binder fiber content and maximum stress in machine direction of silk nonwoven fabrics.

## 2. Elongation at break

Table 4.5 was depicted for the average elongation at break in machine and cross-machine directions. It was seen that the elongation at break in machine direction was higher than that in cross-machine direction. The elongation at break in machine direction was about 30-40%, while in cross-machine direction was 20-30% with a high coefficients of variation. It seemed that the actual areal density affected the elongation at break as much as the binder fiber content (Cusick et al., 1963 ; Marcher, 1991). All samples with similar actual areal density gave no significant difference in the elongation at break.

It was also apparent that the elongation at break of silk nonwoven fabrics were comparable to that of Sontara and Skawol except the elongation at break in cross-machine direction of Sontara which showed a considerably high value.

## 3. Initial Modulus

Initial modulus was obtained from the initial slope of the stress-strain curve. All average initial modulus values only obtained from the machine direction testing in Table 4.6 were computed by the Lloyd analysis computer software. Initial modulus is the resistance to deformation of material, and can use to explain the stiffness. In Figure 4.8, it was obviously seen that increasing bind fiber contents gave the increases in initial modulus. On the other hand, fabrics were stiffer as increasing of PET binder fibers.

Table 4.5 Average elongation at break of the silk nonwoven fabrics in the machine and cross-machine directions.

Samples	Elongation at Break, %			Elongation at Break, %		
	Machine Direction			Cross-machine Direction		
	AVE.	S.D.	%C.V.	AVE.	S.D.	%C.V.
SONTARA <sup>(1)</sup>	39.5	1.2	3.05	115.9	4.5	4.09
SKAWOL <sup>(2)</sup>	24.4	2.3	9.73	23.4	1.8	7.93
40PE30	32.6	2.2	6.67	22.7	8.3	36.56
60PE30	33.4	2.4	7.07	18.3	5.6	30.06
40PET30	39.5	1.9	4.85	26.9	3.7	13.75
60PET20	41.9	2.2	5.21	20.9	5.7	27.27
60PET25	41.4	3.0	7.31	22.7	2.9	12.77
60PET30	33.2	4.5	13.52	20.3	4.1	20.20
60PET35	39.1	4.2	10.88	20.7	4.9	23.67

(1), (2) : The available nonwoven products in the market (Atsawahem and Wiraset, 1993).

Table 4.6 Average initial modulus of the silk nonwoven fabrics.

Samples	Initial Modulus, N/mm <sup>2</sup>		
	AVE.	S.D.	%C.V.
40PE30	25.24	2.63	10.44
60PE30	19.59	5.66	28.93
40PET30	19.04	4.13	21.72
60PET20	13.75	1.32	9.64
60PET25	20.89	2.81	13.46
60PET30	30.19	1.36	4.49
60PET35	34.56	3.99	11.56

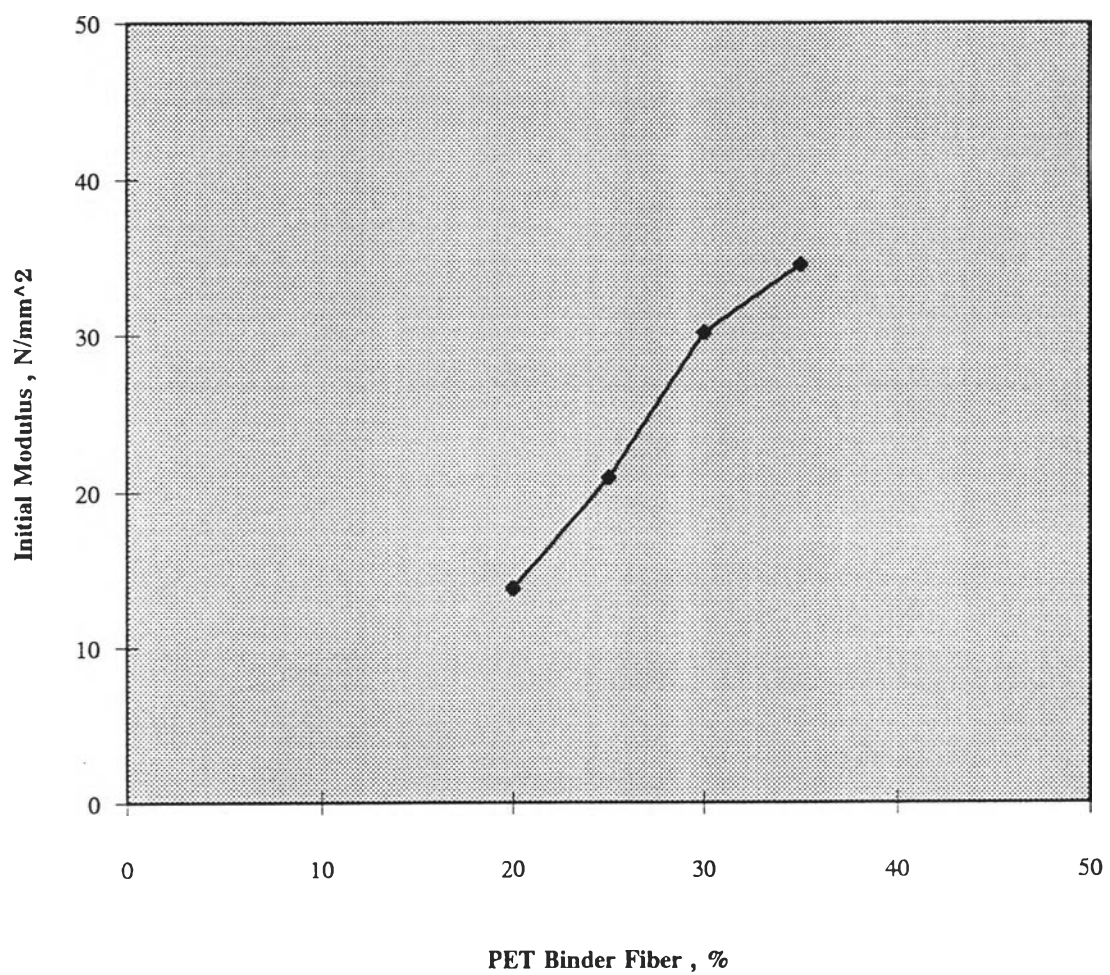


Figure 4.8 Correlation between PET binder fiber content and initial modulus of silk nonwoven fabrics.





## Tear Strength

The average tear strength of the nonwoven fabrics by the pendulum method and single tongue method according to JIS L 1085 in the machine direction were shown in Table 4.7. The maximum load applied to tear the specimens was a indication of the tear strength. Only the machine direction testing was reported because the tearing direction of the cross-machined direction specimens tended to run into the machine direction due to the parallelized arrangement of the fibers. The two methods gave the results in the similar tendency.

The correlation of PET binder fiber contents against the maximum load used to tear the fabrics by two methods in accordance with JIS L 1086-1992, was shown in Figure 4.9 and Figure 4.10. It was obvious that higher percentages of binder fiber resulted in stronger tear strength of silk nonwoven fabrics. However, the coefficients of variation of maximum load by the single tongue method were higher than that of pendulum method. In comparison with Skawol, except 60PET20 silk nonwoven fabrics were stronger in tear strength, but Sontara again offered a considerably high value.

Table 4.7 Average tear strength of the silk nonwoven fabrics by pendulum method and single tongue method in the machine direction.

Samples	Maximum Load, N					
	Pendulum Method			Single Tongue Method		
	AVE.	S.D.	%C.V.	AVE.	S.D.	%C.V.
SONTARA <sup>(1)</sup>	n.a.	-	-	10.47	0.70	6.67
SKAWOL <sup>(2)</sup>	n.a.	-	-	2.66	0.28	10.36
40PE30	10.41	0.40	3.89	4.95	0.63	12.74
60PE30	10.00	0.84	8.40	4.66	0.62	13.36
40PET30	10.49	0.66	6.31	3.04	0.45	14.82
60PET20	11.44	0.87	7.62	1.18	0.20	16.84
60PET25	12.20	1.09	8.97	4.12	0.25	6.15
60PET30	12.32	0.77	6.22	4.51	0.68	15.20
60PET35	13.52	1.62	11.98	5.24	0.73	13.97

(1), (2) : The available nonwoven products in the market (Atsawahem and Wiraset, 1993).

n.a. : not available (Atsawahem and Wiraset, 1993).

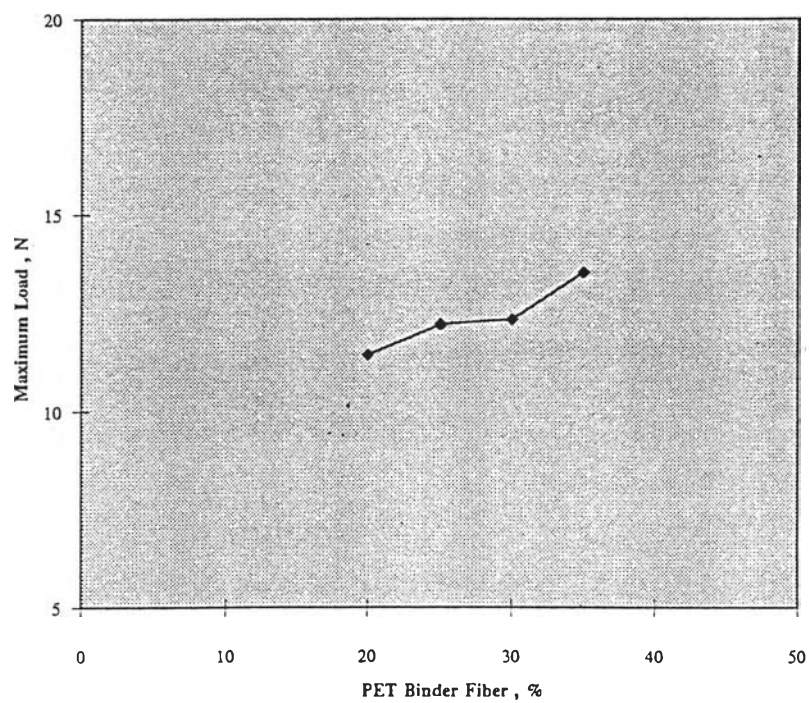


Figure 4.9 Correlation between PET binder fiber content and maximum load of tear strength by pendulum method of silk nonwoven fabrics.

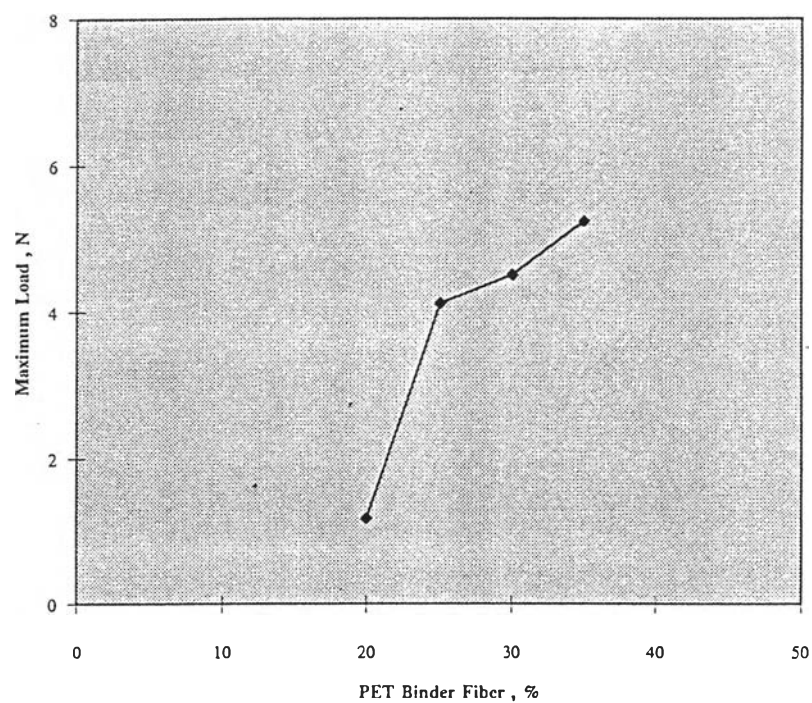


Figure 4.10 Correlation between PET binder fiber content and maximum load of tear strength by single tongue method of silk nonwoven fabrics.

### Effects of Polyester Contents

The conclusive physical and mechanical properties of silk nonwoven fabrics used polyester fibers as binders which contents were varied to 20%, 25%, 30%, and 35% at areal density  $60 \text{ g/m}^2$ , were shown in Table 4.8. It was found that at 20% of polyester fibers, the fabrics were softer relating to their low initial modulus than other percentages, but tensile strength and tear strength were considerable low. Although at 35% of polyester fibers, the fabrics have highest strength in comparison with the others, they were rather stiff including dull appearance, and low moisture regain. Thus the fabrics containing 25-30% of polyester fibers have moderate strength and softness that could be compared to Sontara and Skawol.

Table 4.8 Physical and mechanical properties of the silk nonwoven fabrics produced from waste silk blended with polyester fibers.

PROPERTIES	60PET20	60PET25	60PET30	60PET35
Appearance	Yellowish white, luster, soft hand			
Thickness, mm	0.378	0.345	0.421	0.327
coefficient of variation,%	9.62	8.04	10.38	9.15
Areal density, g/m <sup>2</sup>	61.67	64.53	73.97	65.89
Moisture regain, %	3.19	3.16	3.02	2.68
Air permeability, cm <sup>3</sup> /s /cm <sup>2</sup>	> 160			
Tensile strength, N/mm <sup>2</sup>				
Machine direction	4.26	7.21	10.21	13.69
Cross-machine direction	0.27	0.31	0.90	0.61
Elongation at break, %	41.9	41.44	33.2	39.1
Initial modulus, N/mm <sup>2</sup>	13.75	20.89	30.19	34.56
Tear strength, N				
Pendulum method	11.44	12.20	12.32	13.52
Single tongue method	1.18	4.12	4.51	5.24