CHAPTER V

RESULT AND DISCUSSION

In this chapter, the experimental results of the coating film properties will be discussed. The variables in this work which are the effects of three factors, hydroxyl value (OHV), molecular weight (MW) in term of the catalyst and acid value (AV) refered to Table 4.2 are discussed by varying the formulas of monomers in polyol synthesis step. Based on the experimental design concept, seven factors consisting of three main effects and four interaction effects as shown in Table 3.1 and 3.2 are analyzed in this research. The difference between two hardeners, IPDI and NBDI[1] will also be compared by changing the hardener mixed with polyol before coating on the substrate and curing to produce the coating films. The discussion is made according to the methods of film tests.

1.Pencil hardness Test

Table 5.1 *Pencil hardness.*

Sample	Result	Sample	Result
3I	F	***3N***	H
4I	F	***4N***	H
5I	F	***5N***	H
6I	F	***5N***	H
7I	H	***7N***	H
8I	H	***8N***	H
9I	H	***9N***	H
10I	H	***10N***	H

[1] IPDI is IPDI isocyanurate and NBDI is NBDI isocyanurate.

For IPDI, the hydroxyl value (OHV) is the only one factor that affects the hardness. The samples in the first group (3I-6I), which have low hydroxyl value (OHV=60), show the scale F of pencil hardness while the other group (7I-10I), which have high hydroxyl value (OHV=80) have the scale H. So the hardness can be improved by increasing OHV but the change in the amount of catalyst and acid value (AV) can not affect the hardness. For NBDI, the hardness of all samples has the same pencil hardness scale H. Consequently, the variation of three factors cannot affect the hardness.

Comparing the two hardeners mixed with the same polyol, NBDI has higher hardness at low OHV but the same pencil hardness at high OHV. The samples 3I-6I have the pencil hardness F and the samples 3N-6N have the pencil hardness H in low OHV but the samples 7I-10I and 7N-10N have the same pencil hardness H at in high OHV.

2.Bending Test

Table 5.2 *Bending Test*

Sample	Result	Sample	Result
3I	#2	***3N***	#2
4I	#2	***4N***	#2
5I	#2	***5N***	#2
6I	#2	***6N***	#2
7I	#3	***7N***	#2
8I	#2	***8N***	#2
9I	#2	***9N***	#2
10I	#2	***10N***	#2

In this test, the bending property is specified in the number of rod which the plate is folded around the rod. The number of rod is in the range of #2 to #6. Most of the sixteen samples have the same results #2, the smallest size of rod, but only sample 7I has #3 but the difference is small. So it can be said that most of the samples produce the best result in bending property.

3. Dupont impact test

The impact resistance is an ability to undergo deformation at very rapid rate without cracking. It is used as an index in studying flexibility property.

From Figure 5.1, NBDI sample has much higher Dupont impact than IPDI sample in every polyol. In Figure 5.2, the Dupont impact of NBDI sample and IPDI sample calculated from the average Dupont impact of eight polyols with the same hardener are compared. However, for NBDI, the comparison in this property among them is not possible at the condition, 1/2" diameter and 500 g, because most of them (3N, 4N, 5N, 6N and 8N) give the highest value (500 mm) so the more severe condition is necessary. Then the condition, 3/8" diameter and 1 kg, is selected, and the results are shown in Figure 5.3. After that, three factors are studied.

In IPDI, the Dupont impact decreases when hydroxyl value (OHV) and catalyst are increased but it increases when acid value (AV) increases as shown in Figure 5.4, 5.6 and 5.8. Similarly, the Dupont impact of NBDI decreases when the OHV is increased as shown in Figure 5.5. However, the influence of catalyst and AV in Dupont impact of NBDI affects in both positive and negative way which can be seen in Figure 5.7 and 5.9. So there are interaction effects between the three single effects to this property. It means that the Dupont impact of NBDI can be affected if some of the other effects change at the same time.

Following the experimental design principle, the importance of each effect can be determined in quantitative value by calculating from the average value of each effect in low and high level following to the Table 5.3 and 5.4 and then subtracting them. This value (Δ) can represent the significance of each effect as shown in Figure 5.10 and 5.11. For IPDI, the factors that affect the Dupont impact are three single effects, A (OHV), B (Catalyst) and C (AV), and one interaction effect (AB). Unlike IPDI, the dupont impact of NBDI is affected mainly by A (OHV).

By experimental design method, Δ will be changed to statistical value, F_{effect}. This value can estimate the significance of each effect. Compare the F value with F_c coming from F distribution table, it can decide which factor is significant or not. If F_c is less than F_{effect} of that factor it means that factor is significant. The F_{effect} are presented in Table 5.3 and 5.4. In Table 5.3, the significant effects of IPDI are A (OHV), B (Catalyst) and AB (OHV and Catalyst) at 99 % confidence. Unlike IPDI, the significant effects of NBDI are A (OHV), C (AV), AB (OHV and Catalyst), AC (OHV and AV), BC (Catalyst and AV) and ABC (OHV, Catalyst and AV) at 99 % confidence.

Dupont impact resistance of NBDI is much higher than that of IPDI. It might result from the differences in the chemical structure between NBDI and IPDI. Both of them have six carbon atoms ring but one of two isocyanate side chain groups of NBDI has longer chain. So, it can be assumed that NBDI might have free volume in segmental movement of molecular chain more than IPDI thus resulting in higher ability for withstanding impact. In the other word, NBDI can absorb more energy than IPDI in the impact testing.

The most crucial factor in dupont impact of both hardeners is OHV. If OHV is decreased, the dupont impact values are increased. It might be attributed to the higher density in network structure of the film which influences in adding stiffness of the film. Thus the film is easier to crack when it is subjected to impact test. Moreover, catalyst is also the important factor that affects on the quality of the film of IPDI. Increase of the amount of catalyst will decrease molecular weight (MW) and then dupont impact values will be decreased. The more molecular weight, the higher dupont impact value because of higher potential of the films on the withstanding the impact energy.

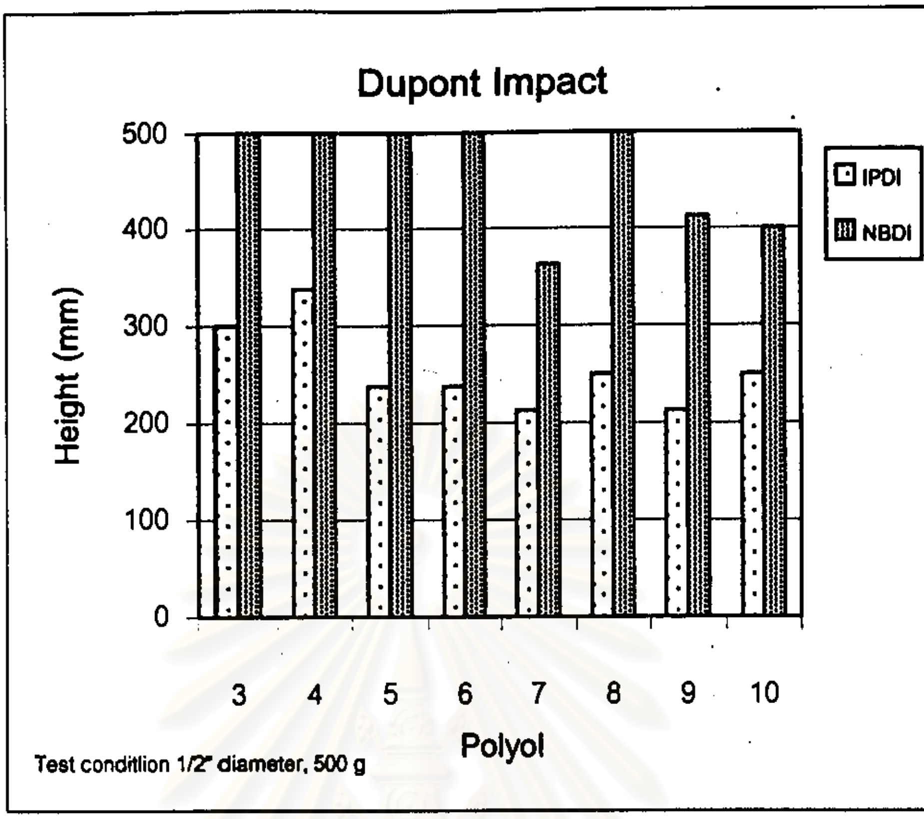

Figure 5.1 Dupont impact of sixteen samples

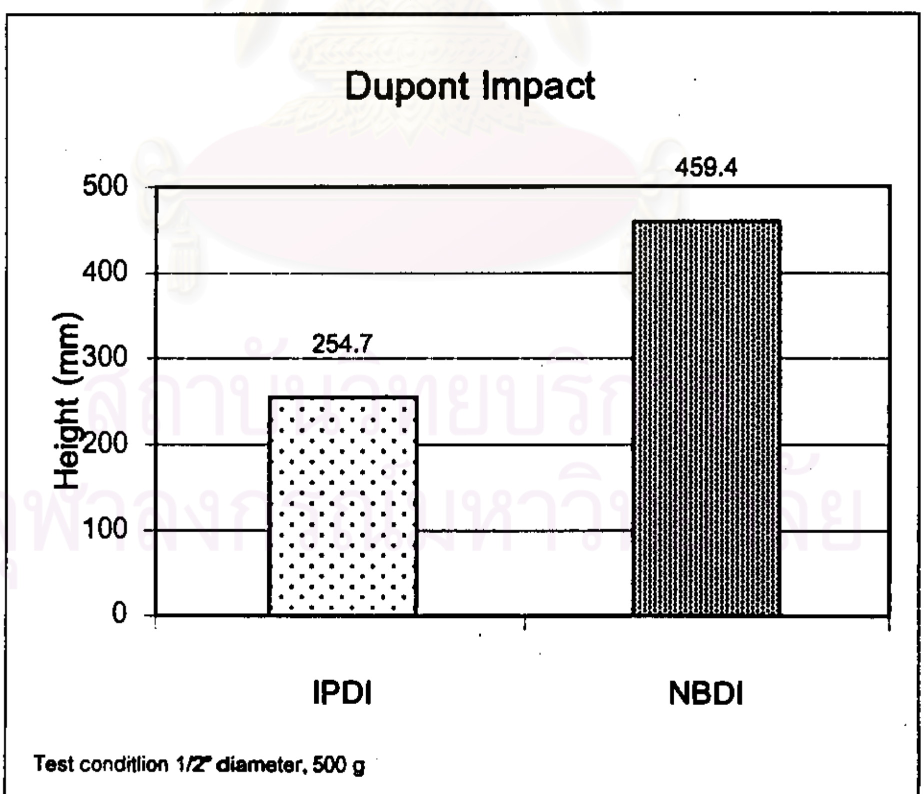

Figure 5.2 Dupont impact of two hardeners

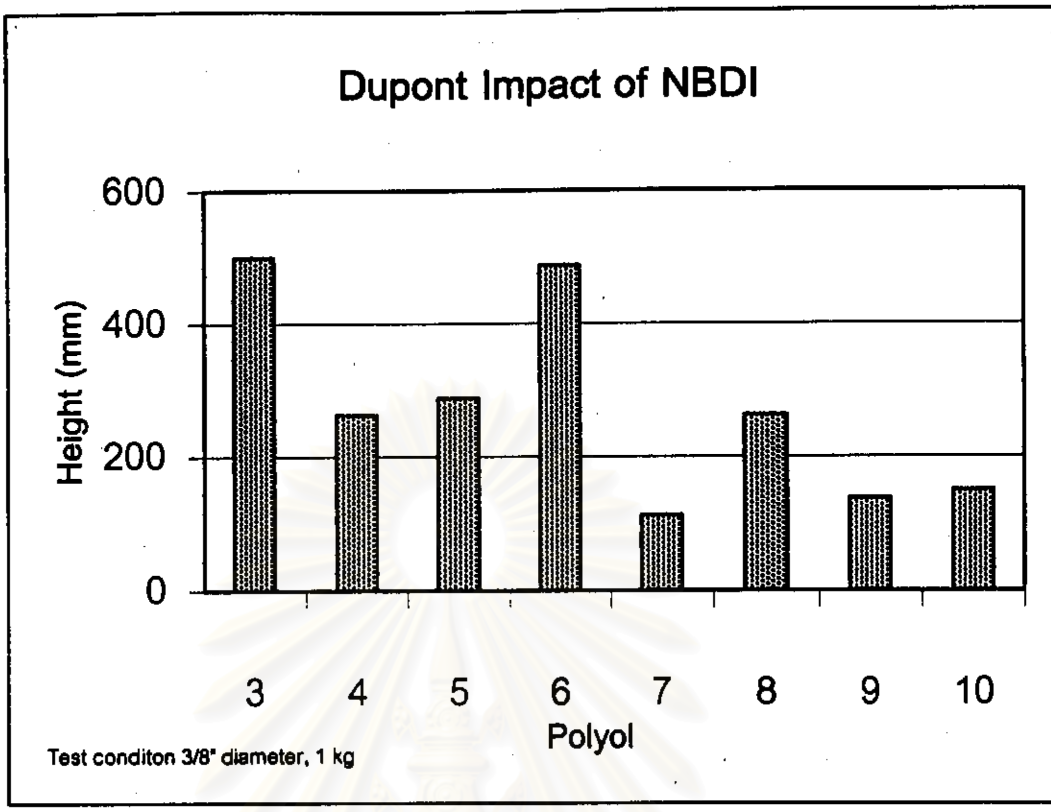

Figure 5.3 Dupont impact of NBDI

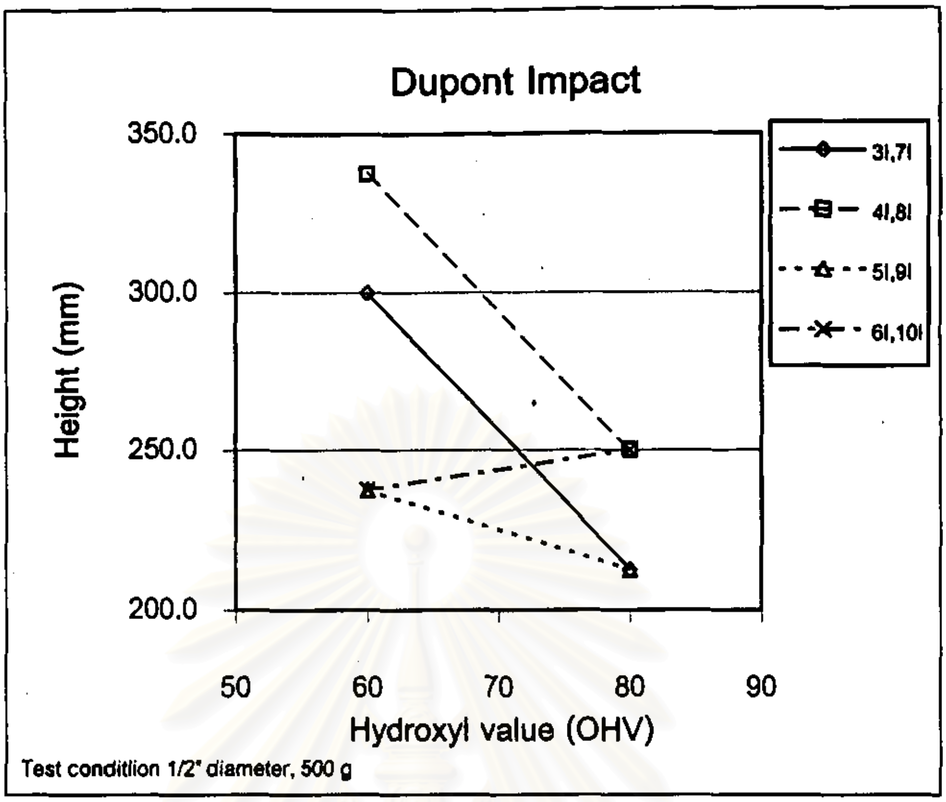

Figure 5.4 Dupont impact vs hydroxyl value of IPDI

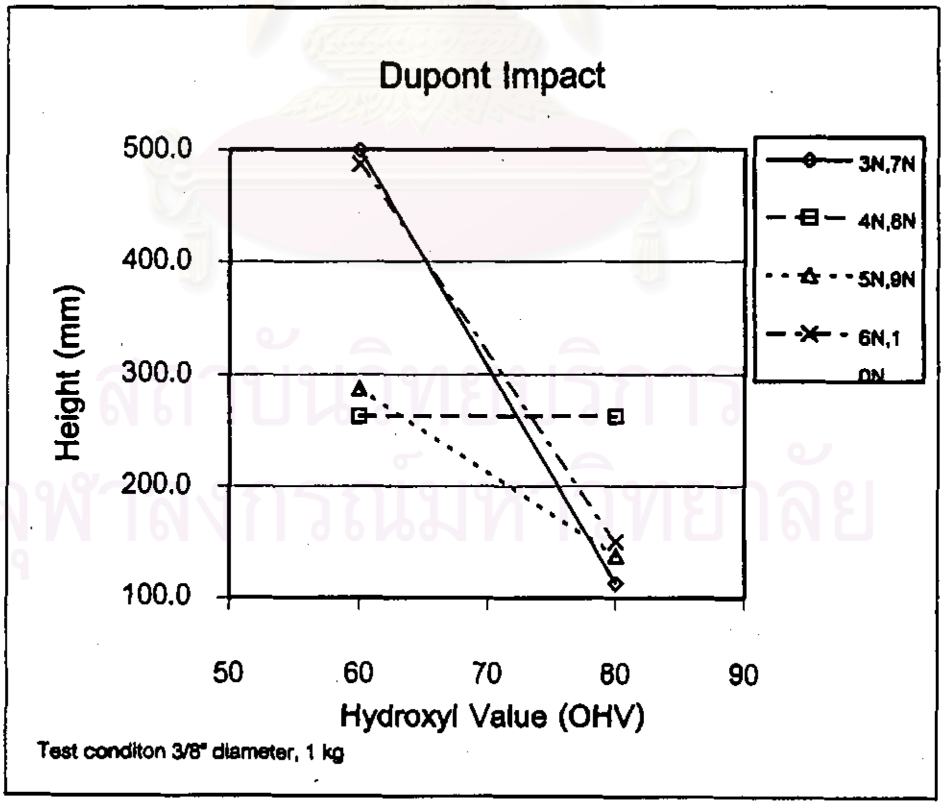

Figure 5.5 Dupont impact vs hydroxyl value of NBDI

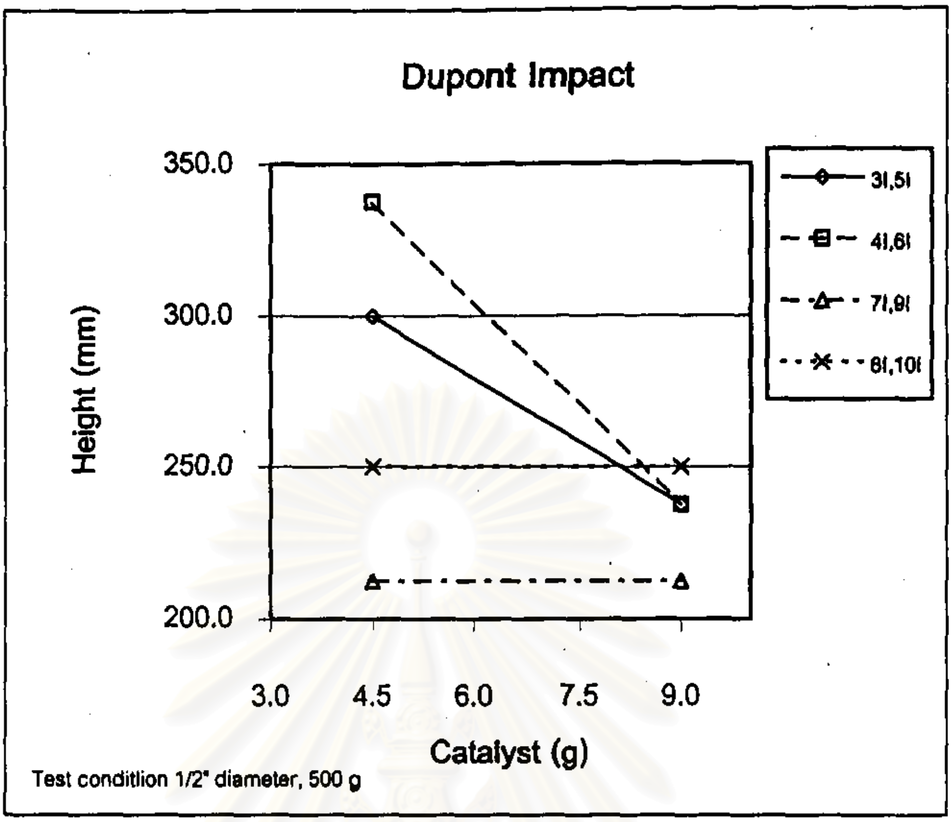

Figure 5.6 Dupont impact vs Catalyst of IPDI

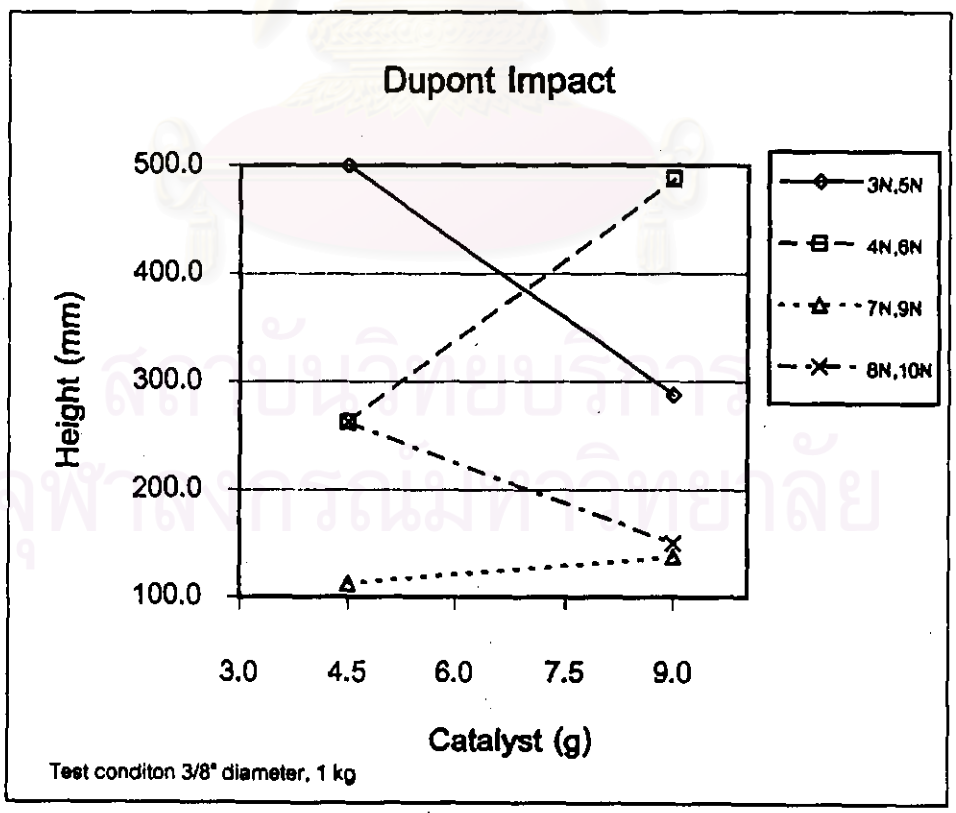

Figure 5.7 Dupont impact vs Catalyst of NBDI

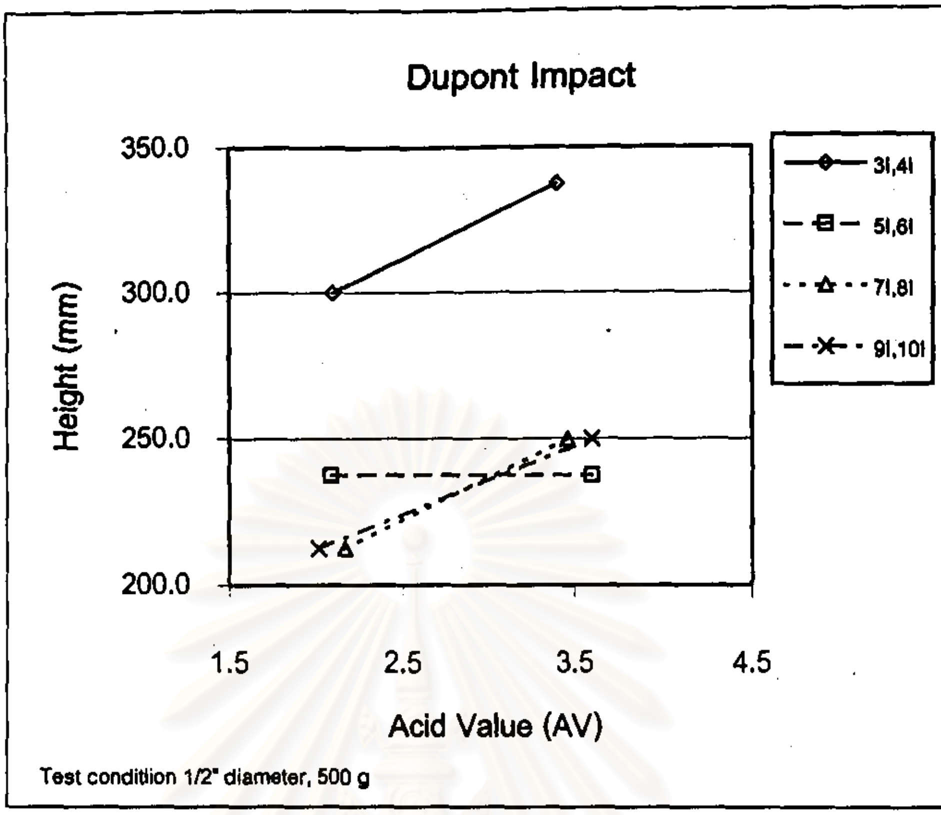

Figure 5.8 Dupont impact vs acid value of IPDI

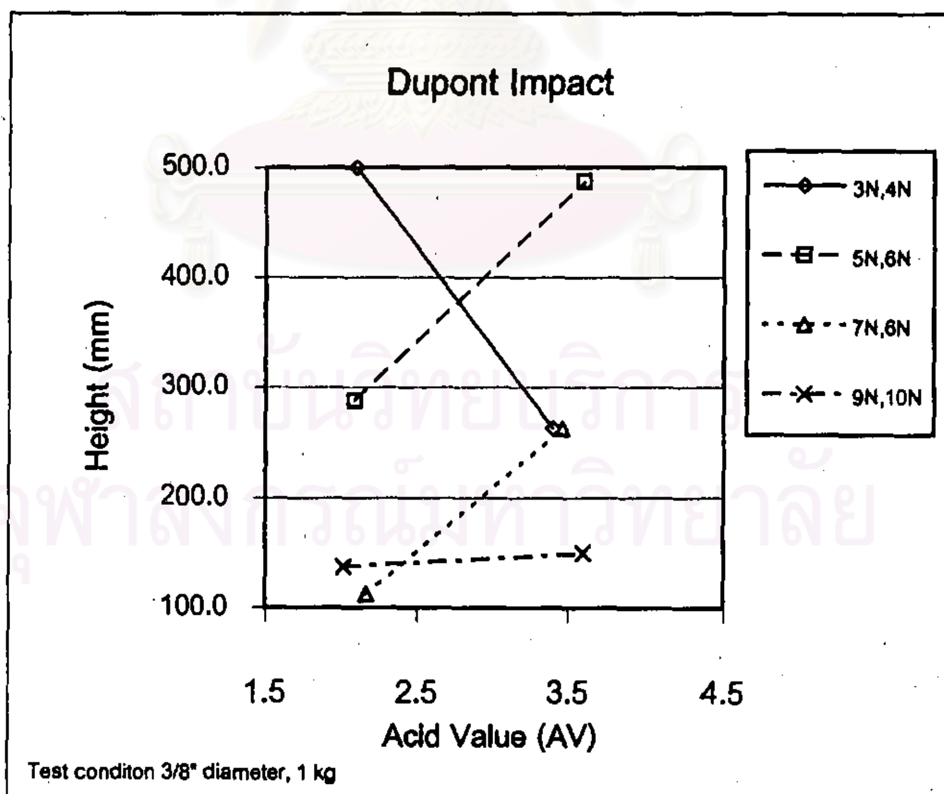

Figure 5.9 Dupont impact vs acid value of NBDI

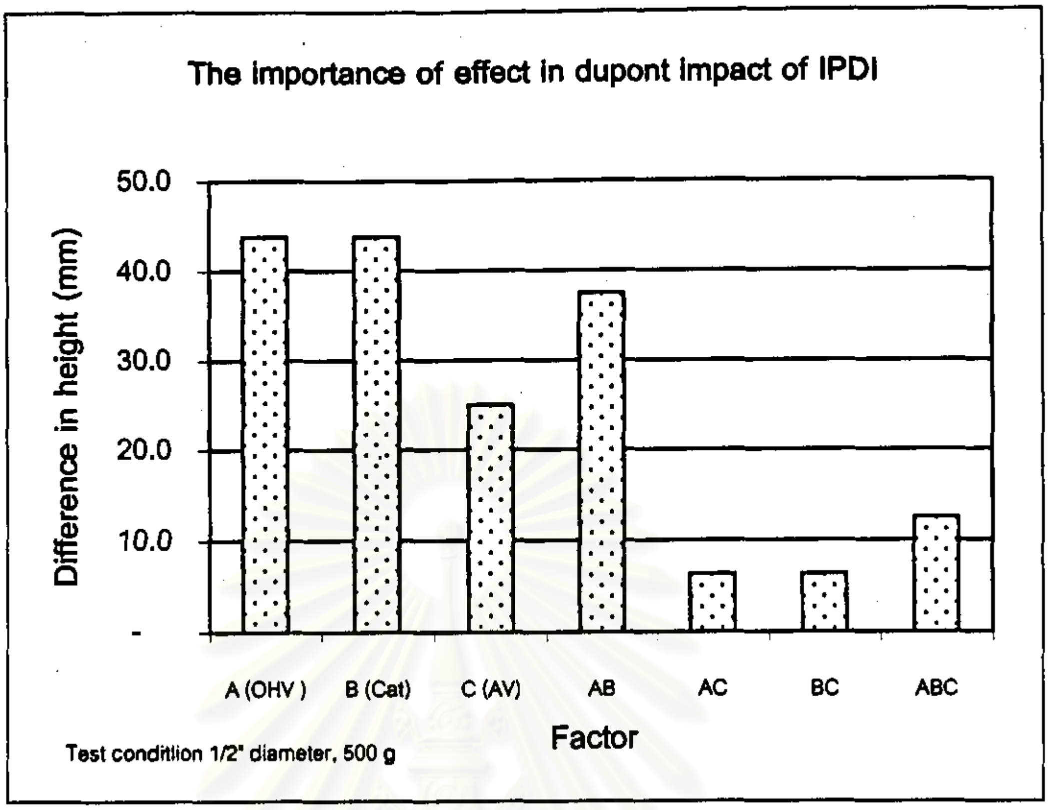

Figure 5.10 The importance of effect in dupont impact of IPDI

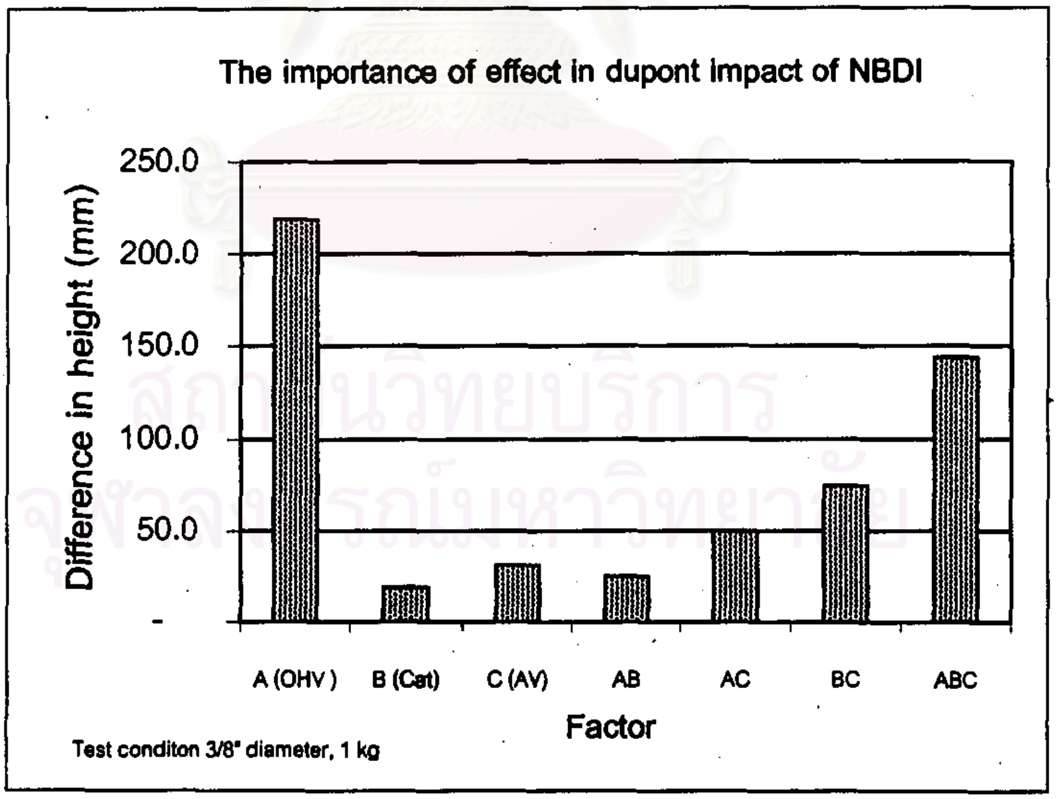

Figure 5.11 The importance of effect in dupont impact of NBDI

***Table 5.3** The importance of effects in dupont impact of IPDI*

	A (OHV)	B (Cat)	C (AV)	AB	AC	BC	ABC
Avg(-)	278.1	278.1	243.8	237.5	253.1	259.4	250.0
Avg(+)	234.4	234.4	268.8	275.0	259.4	253.1	262.5
Δ	-43.8	-43.8	25.0	37.5	6.3	-6.3	12.5
MSB_{effect}	11,484	11,484	3,750	8,438	234	234	938
F_{effect}	27.54	27.54	8.99	20.23	0.56	0.56	2.25

F_C (0.95,1,24) = 4.26 or F_C (0.99,1,24) = 7.82

Note : Test condition 1/2" diameter, 500g

***Table 5.4** The importance of effects in dupont impact of NBDI*

	A (OHV)	B (Cat)	C (AV)	AB	AC	BC	ABC
Avg(-)	384.4	284.4	259.4	287.5	250.0	237.5	346.9
Avg(+)	165.6	265.6	290.6	262.5	300.0	312.5	203.1
Δ	-218.8	-18.8	31.3	-25.0	50.0	75.0	-143.8
MSB_{effect}	287,109	2,109	5,859	3,750	15,000	33,750	123,984
F_{effect}	612.2	4.5	12.5	8.0	32.0	72.0	264.4

F_C (0.95,1,24) = 4.26 or F_C (0.99,1,24) = 7.82

Note : Test condition 3/8" diameter, 1 kg

4.Chemical Resistance Test

The chemical resistance in this work is determined in quantitative values by comparing the values of Dupont impact before and after dipping in acid or base solution. The chemical resistance is reported in the difference of the height in Dupont impact between two conditions (before dipping and after dipping in solution) so it means the greater difference in height of Dupont impact, the less chemical resistance.

In acid resistance, most of IPDI samples give the better performance except 3I and 4I sample as shown in Figure 5.12. Among the NBDI sample, the best are 3N and 4N and the worst is 10N while 3I and 10I are the worst and 5I, 6I, 7I and 8I are the best of IPDI samples.

In base resistance, most of IPDI samples also have more resistance than NBDI samples except sample 7I as presented in Figure 5.13. Comparing among the NBDI samples, the best is 7N and the worst is 6N while 5I and 9I are the best and 4I is the worst of IPDI samples. In general, IPDI has chemical resistance more than NBDI as shown in Figure 5.14 and both of them have acid resistance more than base resistance.

To analyze the three factors, OHV, catalyst and AV, acid resistance is considered first. In Figure 5.15, acid resistance of IPDI is increased with increasing OHV at low catalyst level (3I, 7I and 4I, 8I) but acid resistance is decreased with increasing OHV at high catalyst level (5I, 9I and 6I, 10I) In Figure 5.16, acid resistance of NBDI sample is increased with increasing OHV. In Figure 5.17, the graph is similar to Figure 5.15, the acid resistance of IPDI is increased with increasing catalyst at low OHV level and the acid resistance of NBDI is decreased with increasing catalyst at high OHV level. In Figure 5.18, 5.19 and

5.20, some of line are cross together so it means that there are interaction effect between three factors.

In Figure 5.27 and 5.28, the significance of each effect is demonstrated, and the table 5.5, 5.6 present the numerical value of the significance of each effect. In NBDI, the main effect is A (OHV), BC (catalyst and AV) and ABC (OHV, catalyst and AV). The factors that mainly effect the acid resistance in IPDI samples are interaction effect, AB (OHV and catalyst), AC (OHV and AV) and BC (catalyst and AV).

Concerning base resistance, the resistance of NBDI is increased with increasing in OHV as shown in figure 5.22. For IPDI samples, the base resistance is increased with increasing catalyst and decreased with increasing AV. In the Figure 5.21, 5.23 and 5.25, there are interaction effects between three factors. The importance of each effect is presented in Figure 5.29 and 5.30 ,and its numerical value is presented in table 5.7 and 5.8. In IPDI sample, the main effects are B (catalyst), C (AV) and AB (OHV and catalyst). In NBDI sample the main effect is A (OHV), BC (catalyst and AV) and ABC (OHV, catalyst and AV).

From these results, it can be concluded that the most significant factors of IPDI are Factor AB (OHV and catalyst) while the most important factor of NBDI is A (OHV).

NBDI's films produce less chemical resistance than IPDI's. It might derive from the differences in chemical structures of two hardeners due to the reason that are similar to impact resistance. Consequently, it might believe that the molecular chains of NBDI can pack themselves less densely than that of IPDI due to the longer side chains of isocyanate group of NBDI. As a result, the acid or base can break the chemical bond more readily and penetrate into the coating films. In addition, OHV is the most important factor that affects on this property in both hardeners. The increase in OHV leading to more functionality results in the higher number of junction points in structure, higher density of film so the film gives higher chemical resistance. Furthermore, the amount of catalyst is also one of the significant factor which influences IPDI's film. The increase of the quantity of catalyst, lowering in molecular weight, will increase in chemical resistance because of the higher density of the film.

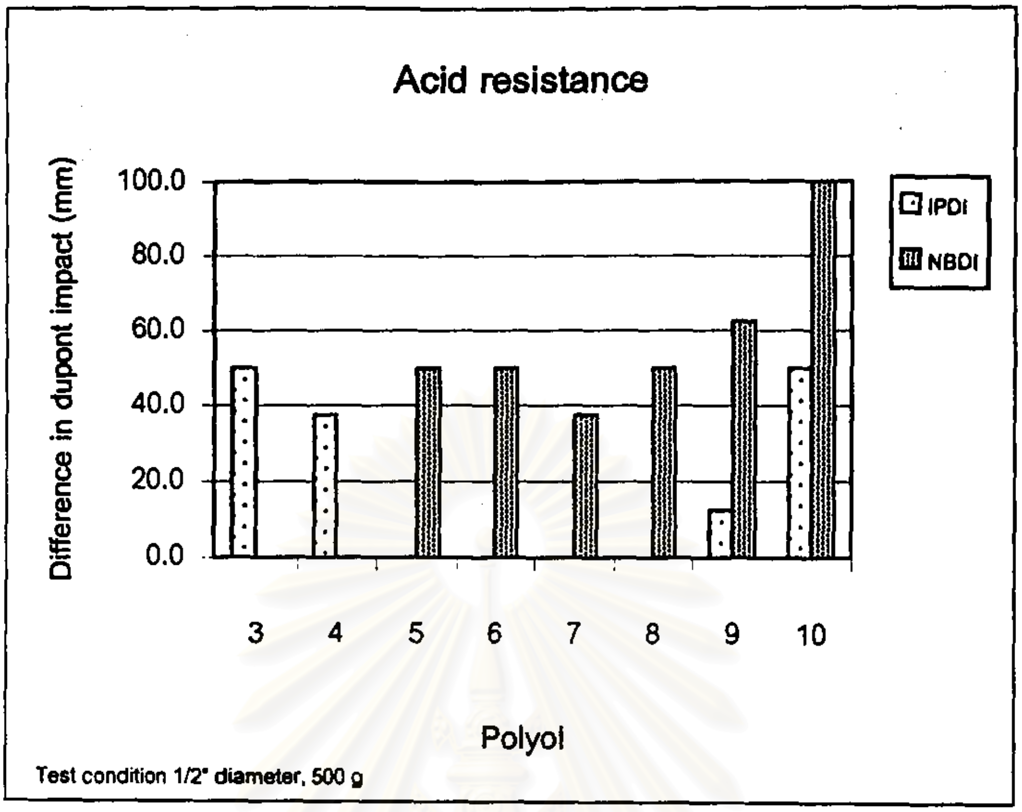

Figure 5.12 Acid resistance of sixteen samples

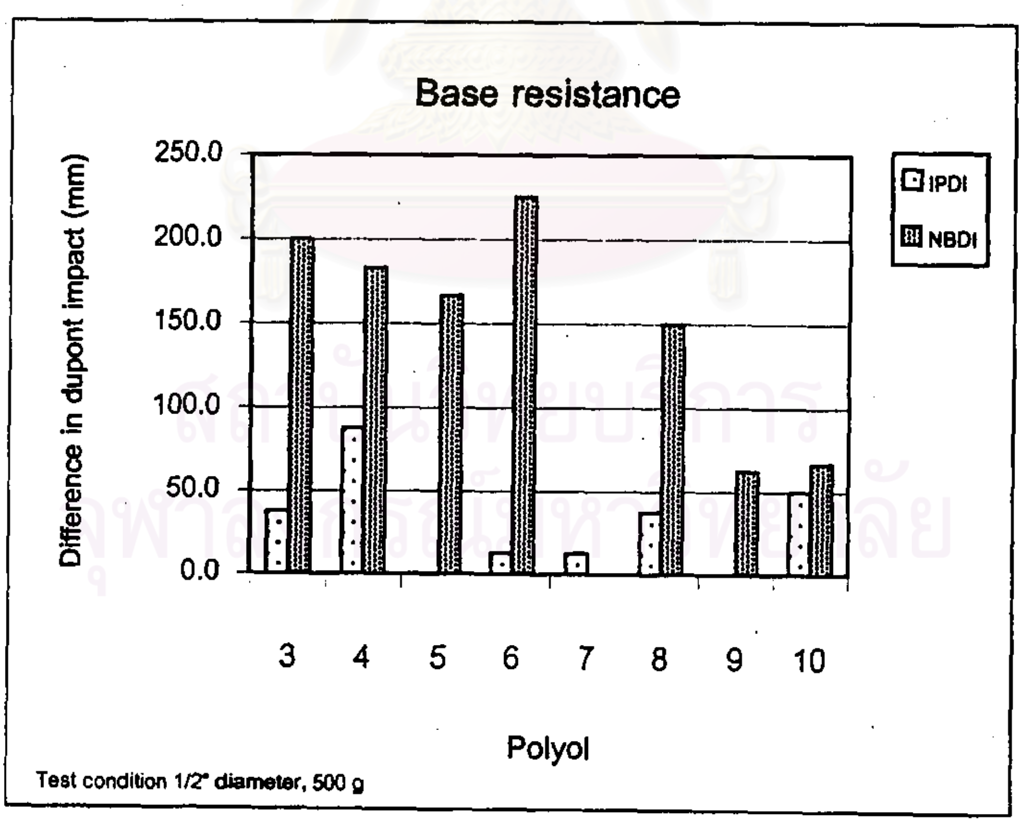

Figure 5.13 Base resistance of sixteen samples

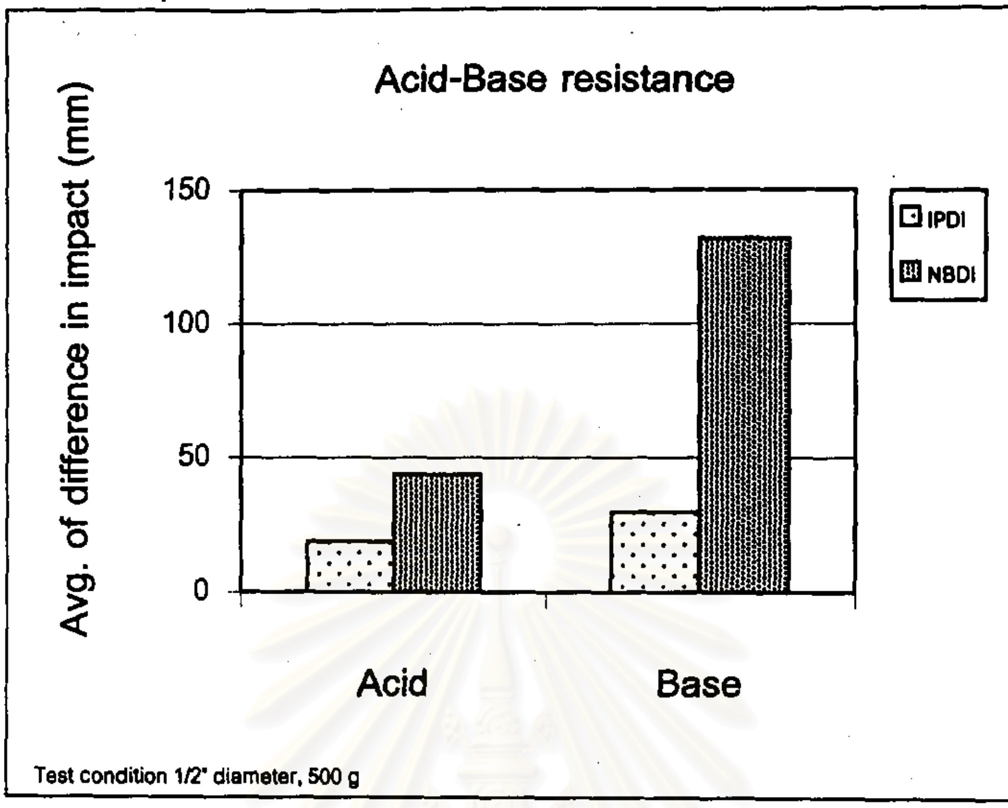

Figure 5.14 Acid-Base resistance of two hardeners

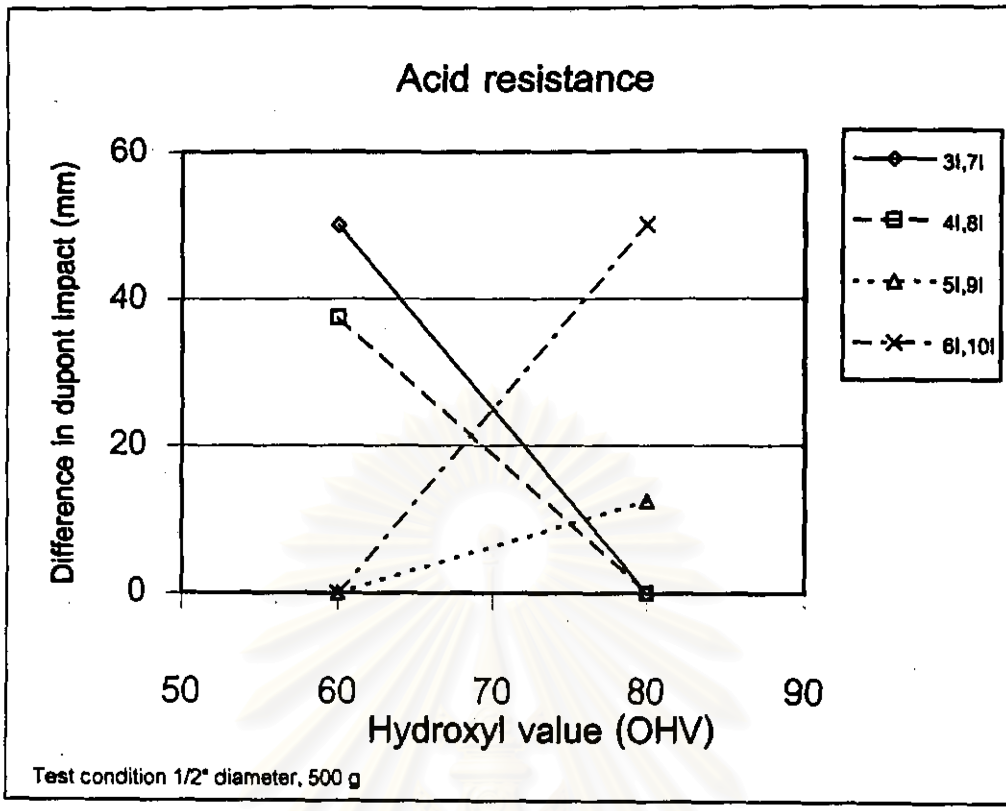

Figure 5.15 Acid resistance vs Hydroxyl value of IPDI

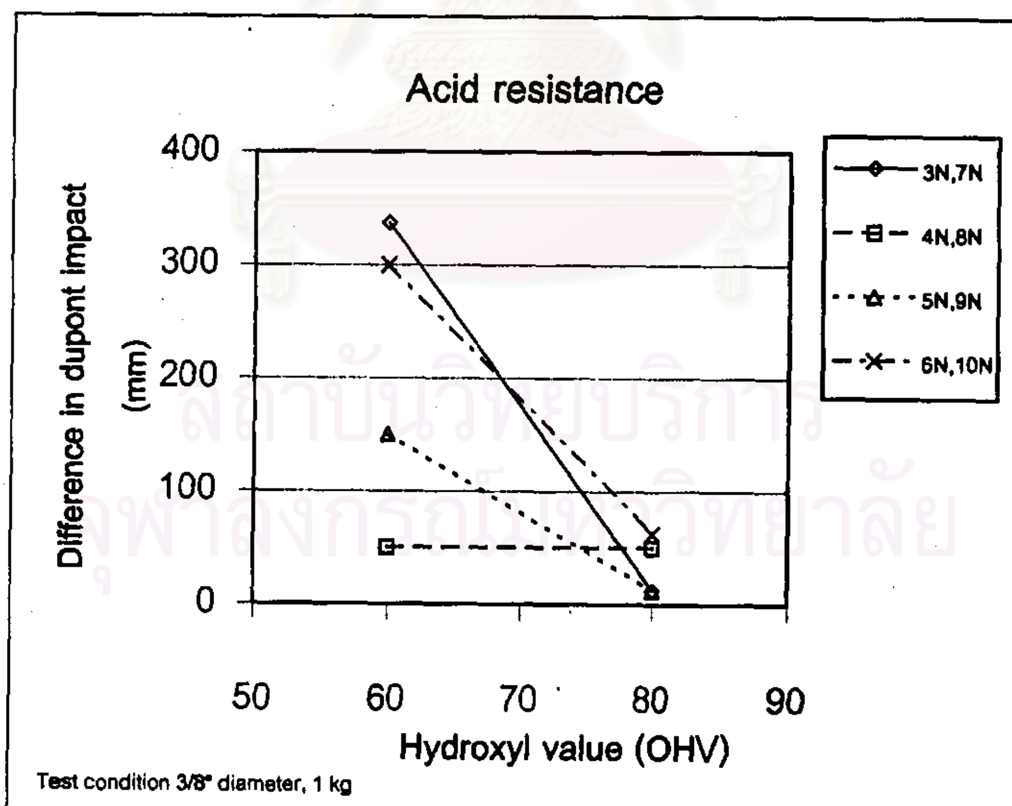

Figure 5.16 Acid resistance vs Hydroxyl value of NBDI

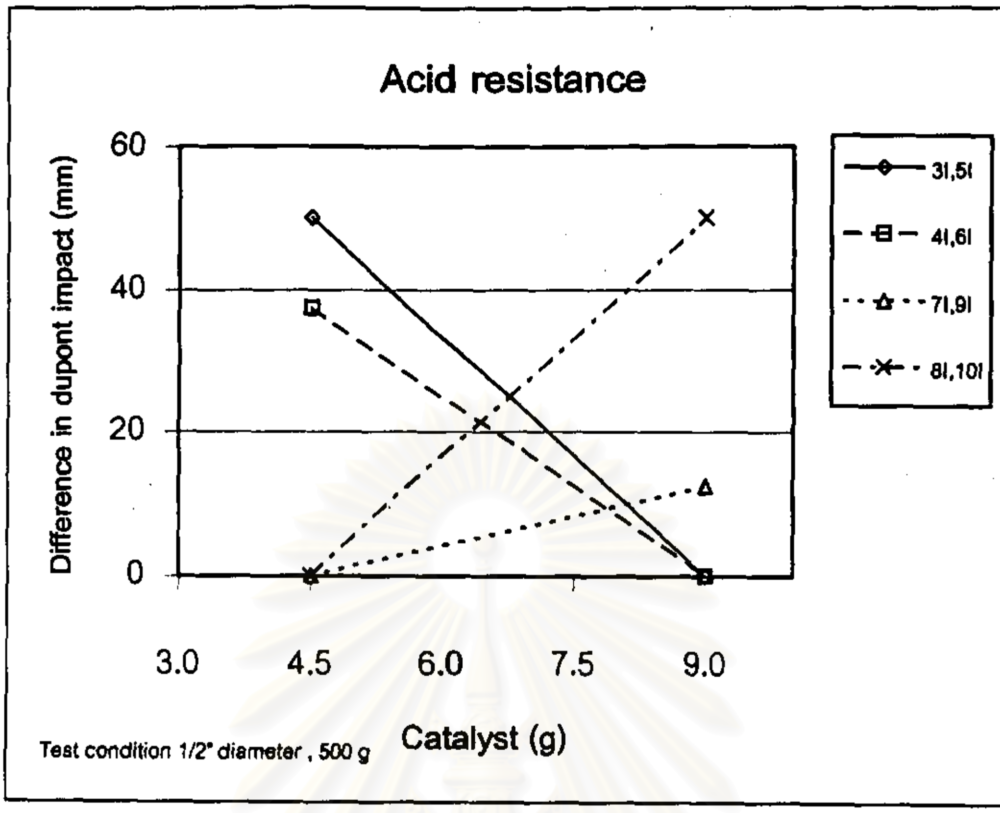

Figure 5.17 Acid resistance vs Catalyst of IPDI

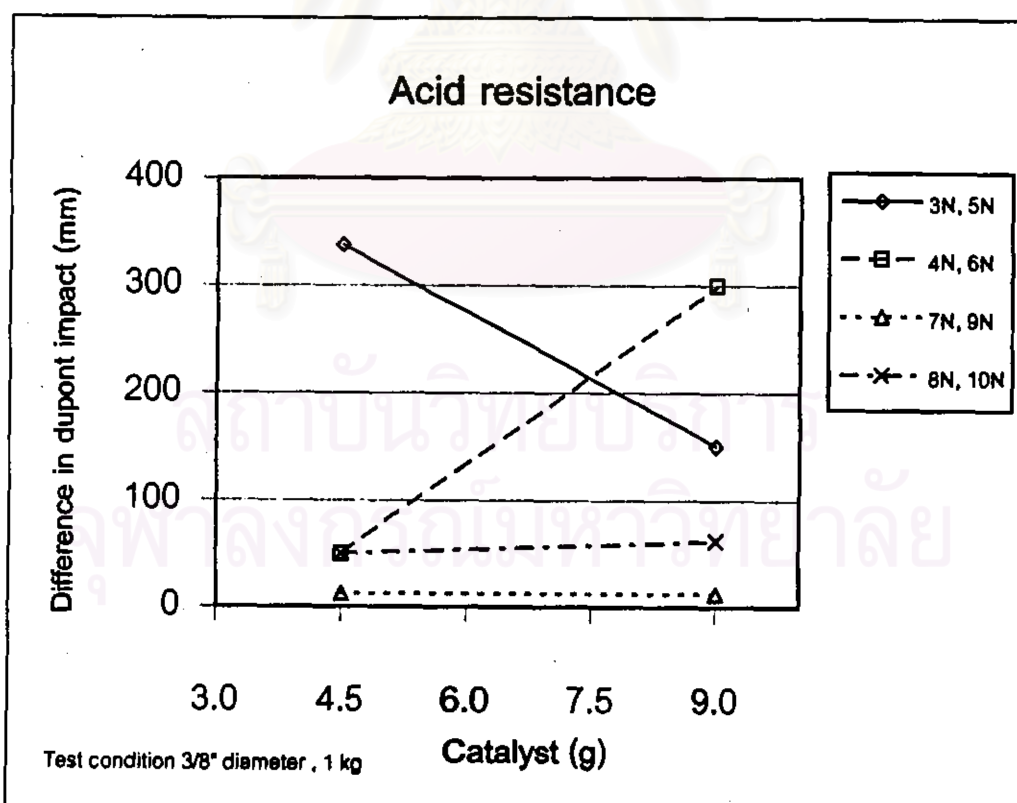

Figure 5.18 Acid resistance vs Catalyst of NBDI

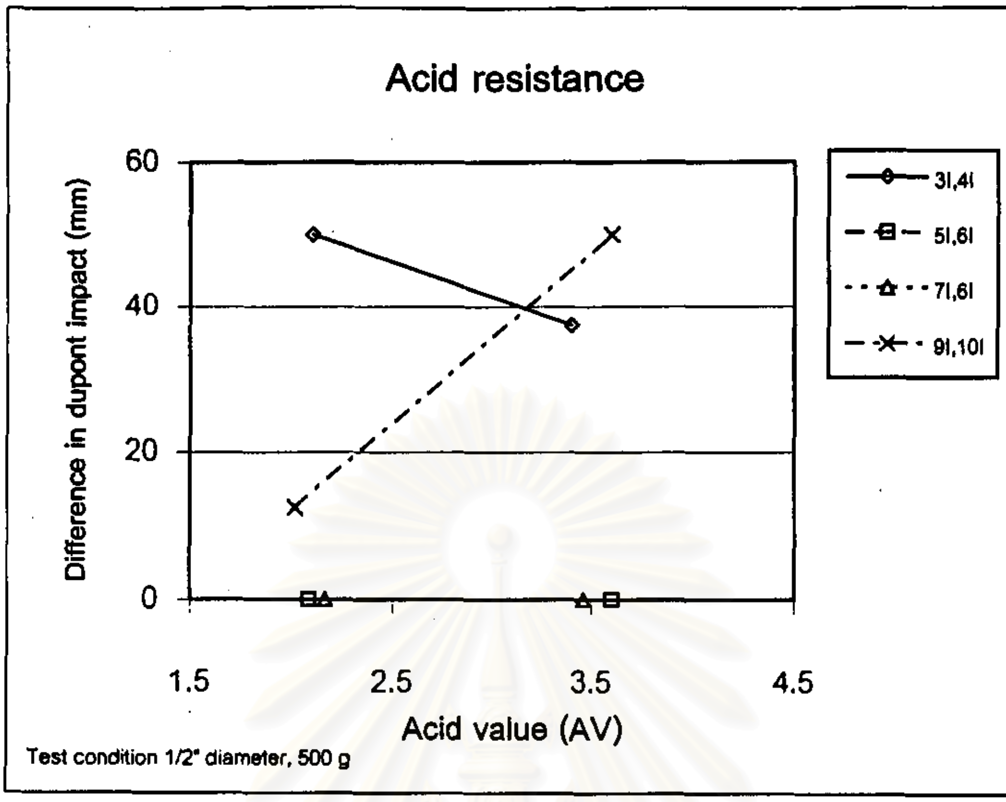

Figure 5.19 Acid resistance vs acid value of IPDI

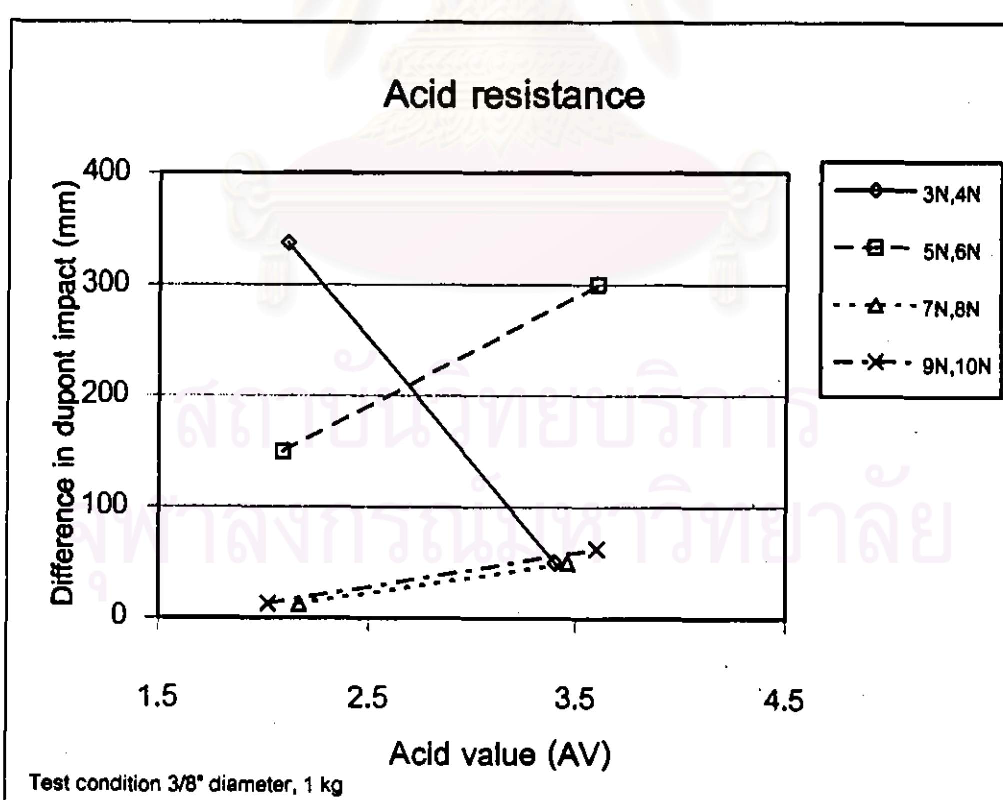

Figure 5.20 Acid resistance vs acid value of NBDI

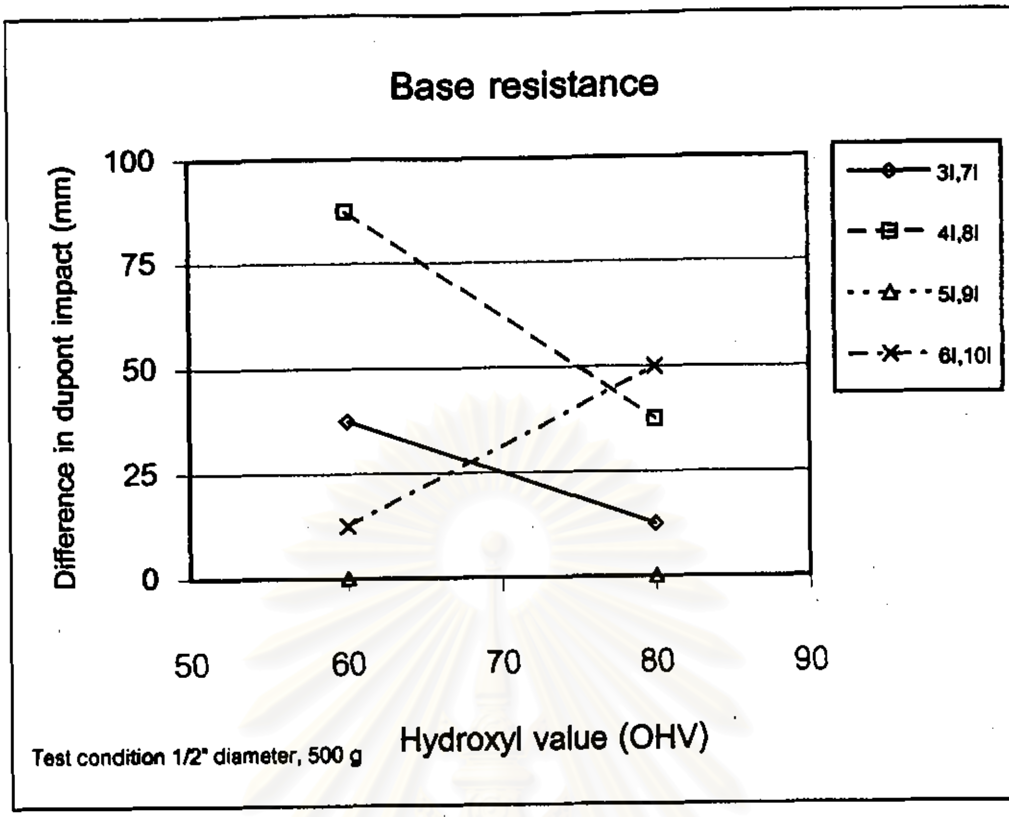

Figure 5.21 Base resistance vs Hydroxyl value of IPDI

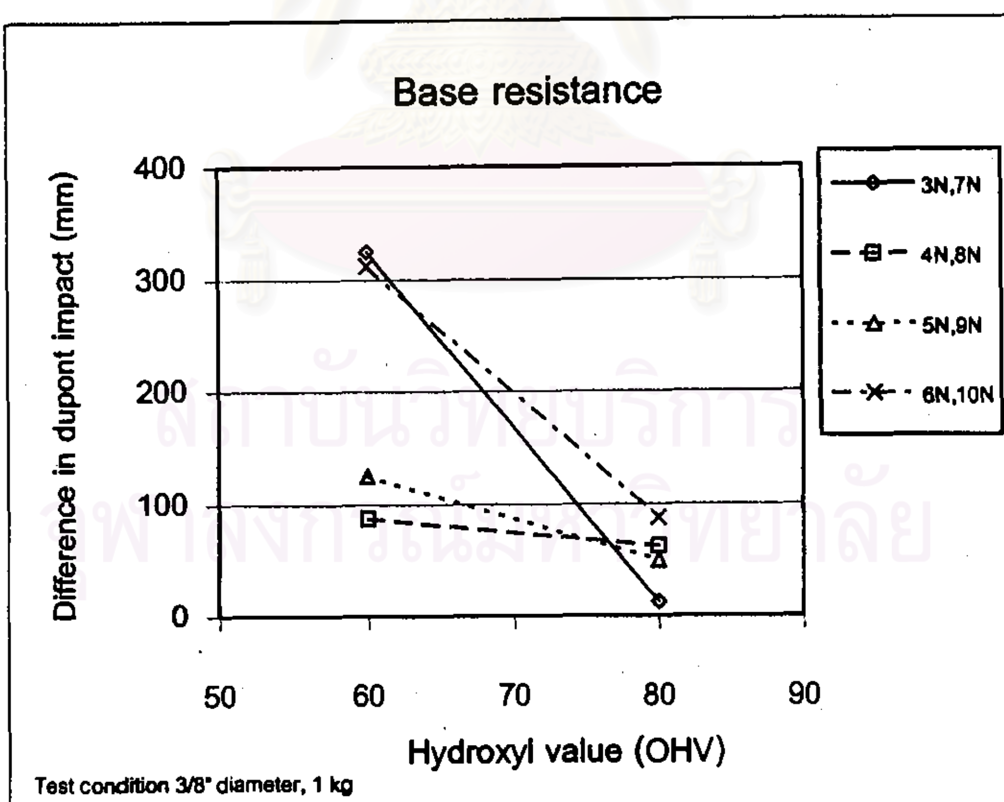

Figure 5.22 Base resistance vs Hydroxyl value of NBDI

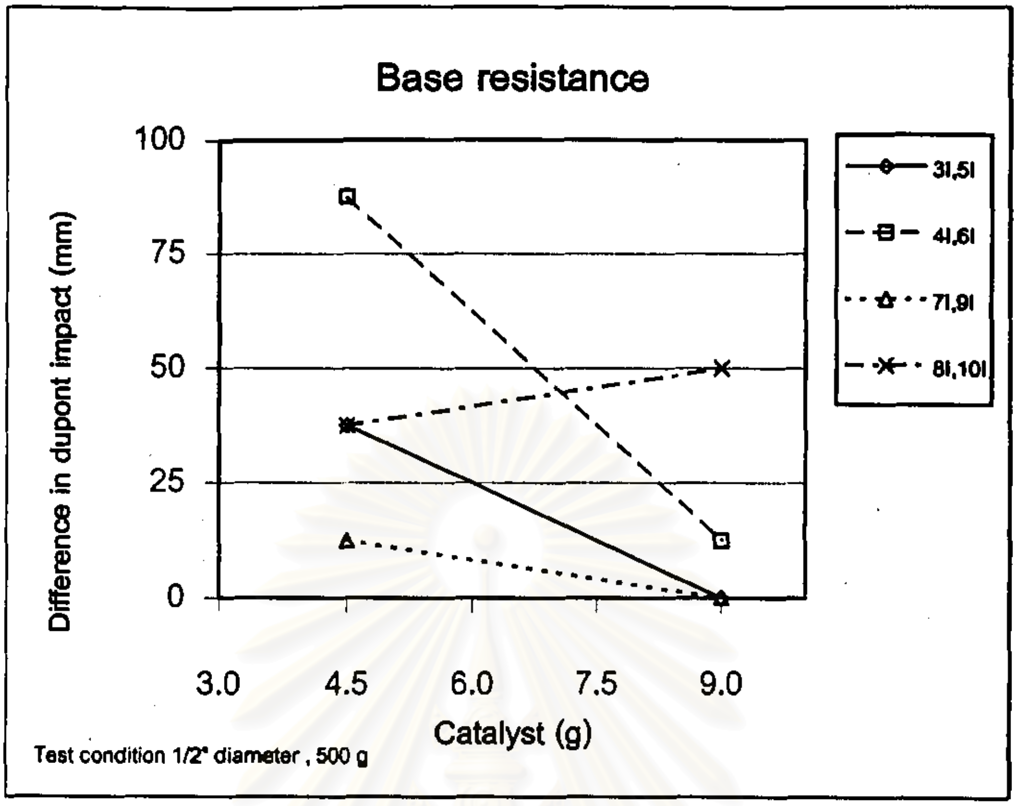

Figure 5.23 Base resistance vs Catalyst of IPDI

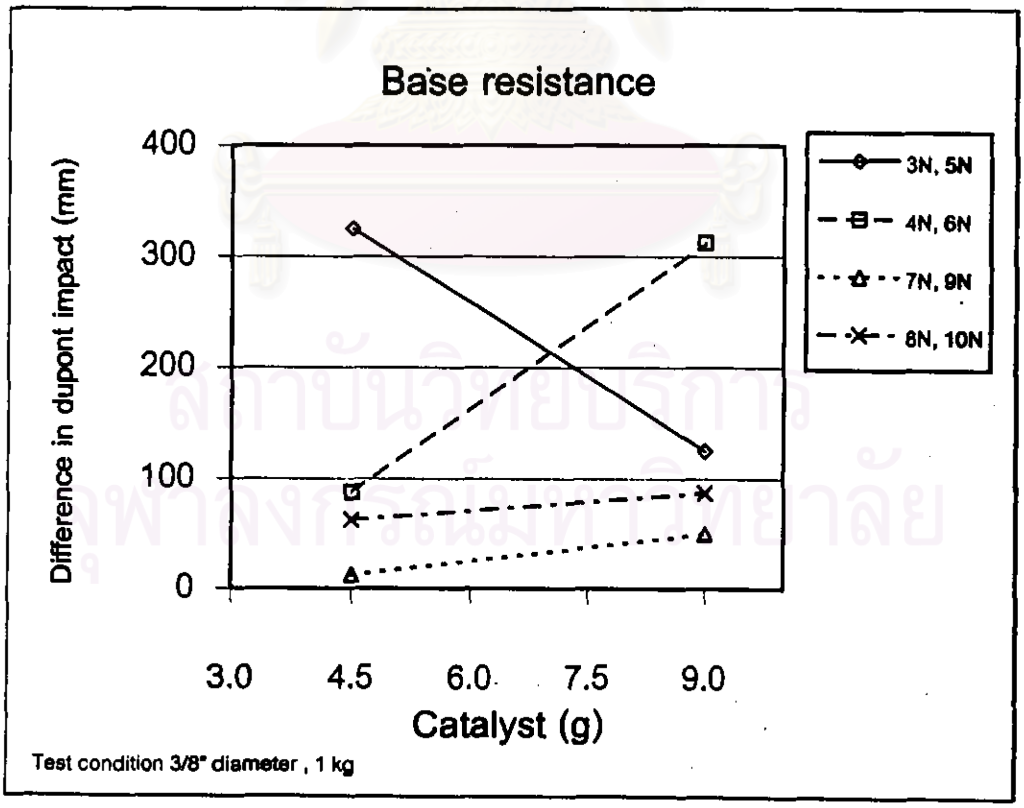

Figure 5.24 Base resistance vs Catalyst of NBDI

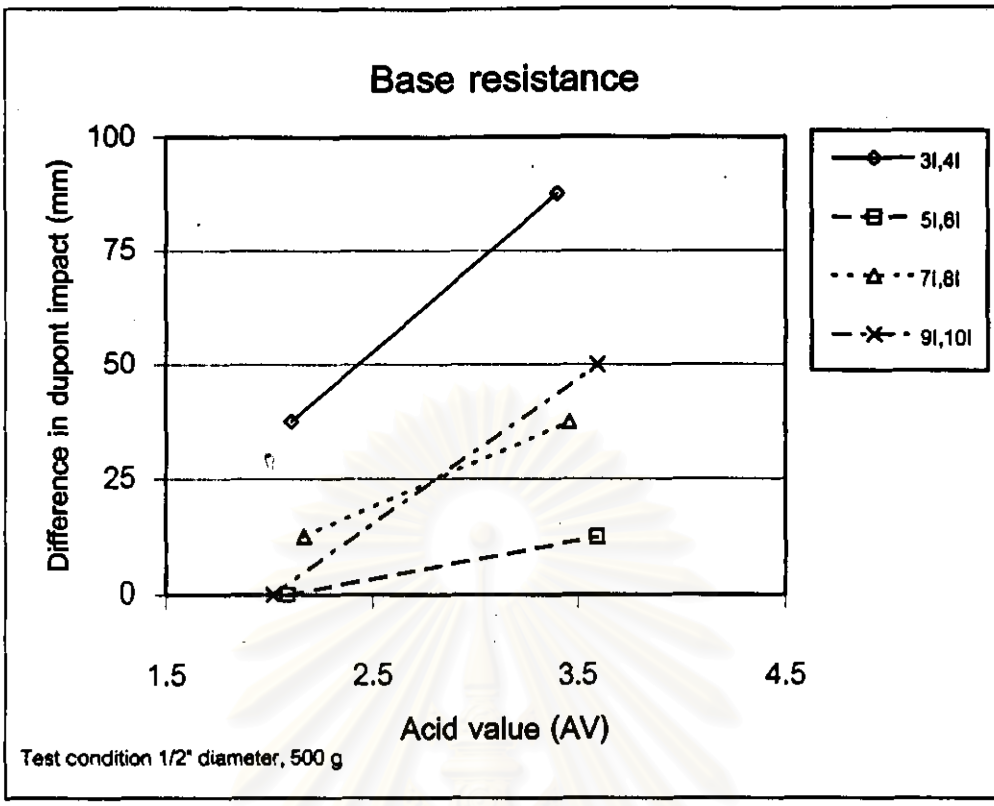

Figure 5.25 Base resistance vs acid value of IPDI

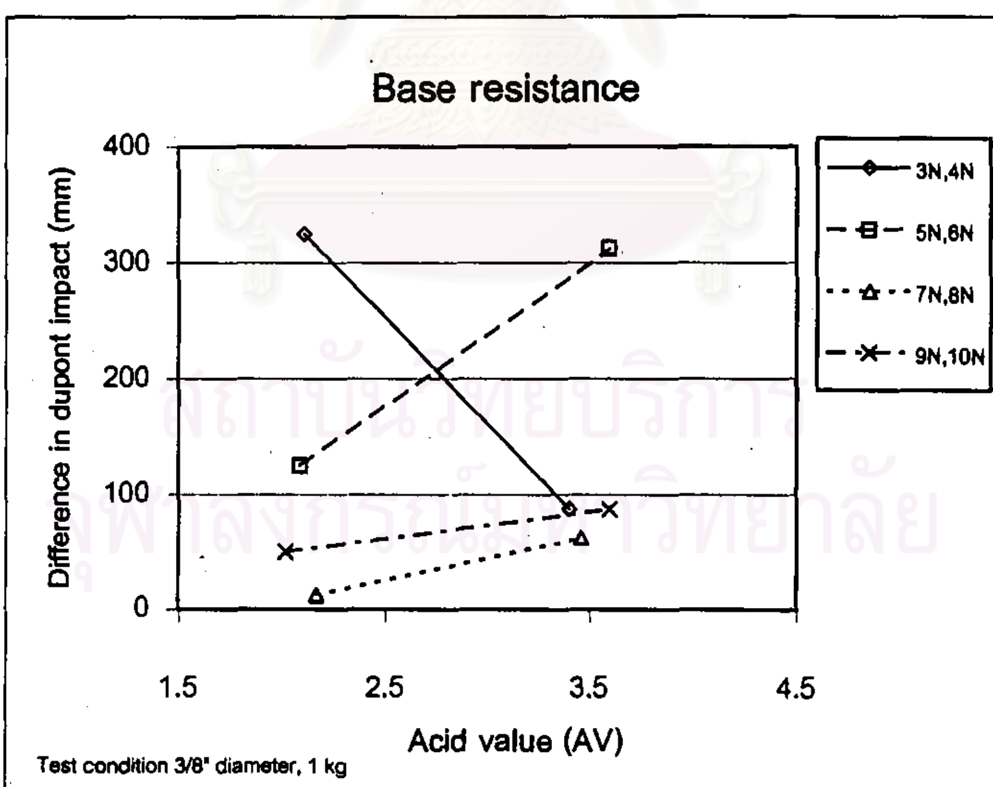

Figure 5.26 Base resistance vs acid value of NBDI

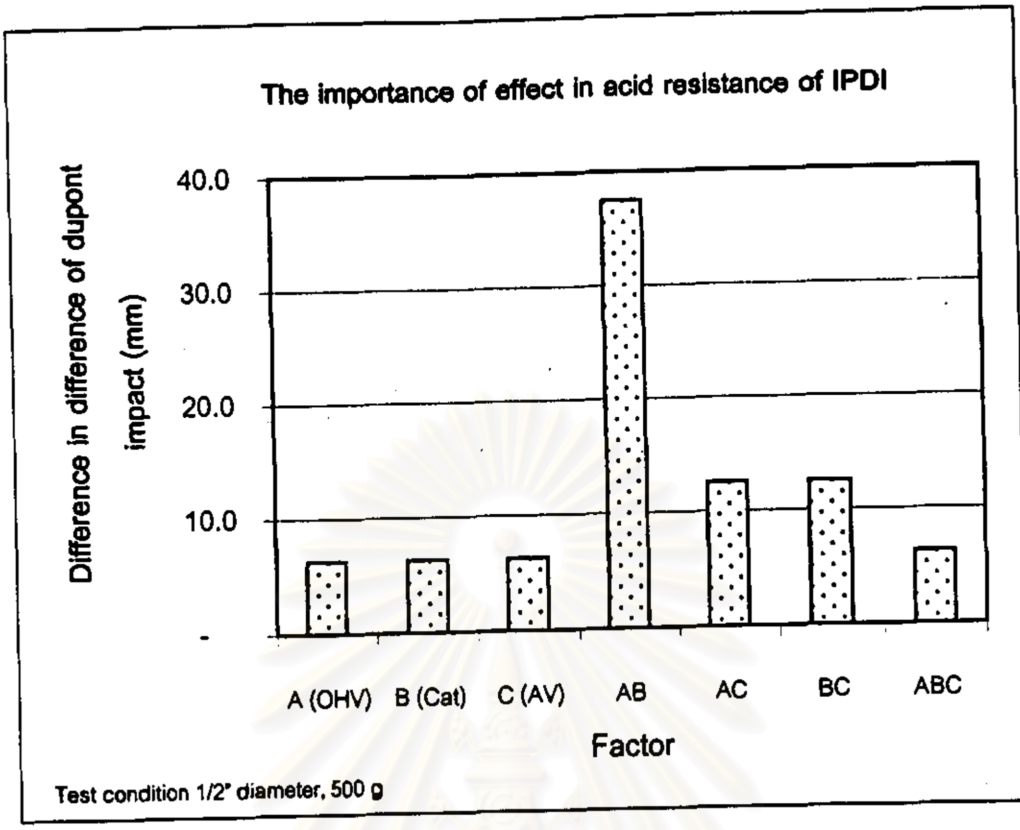

Figure 5.27 The importance of effect in acid resistance of IPDI

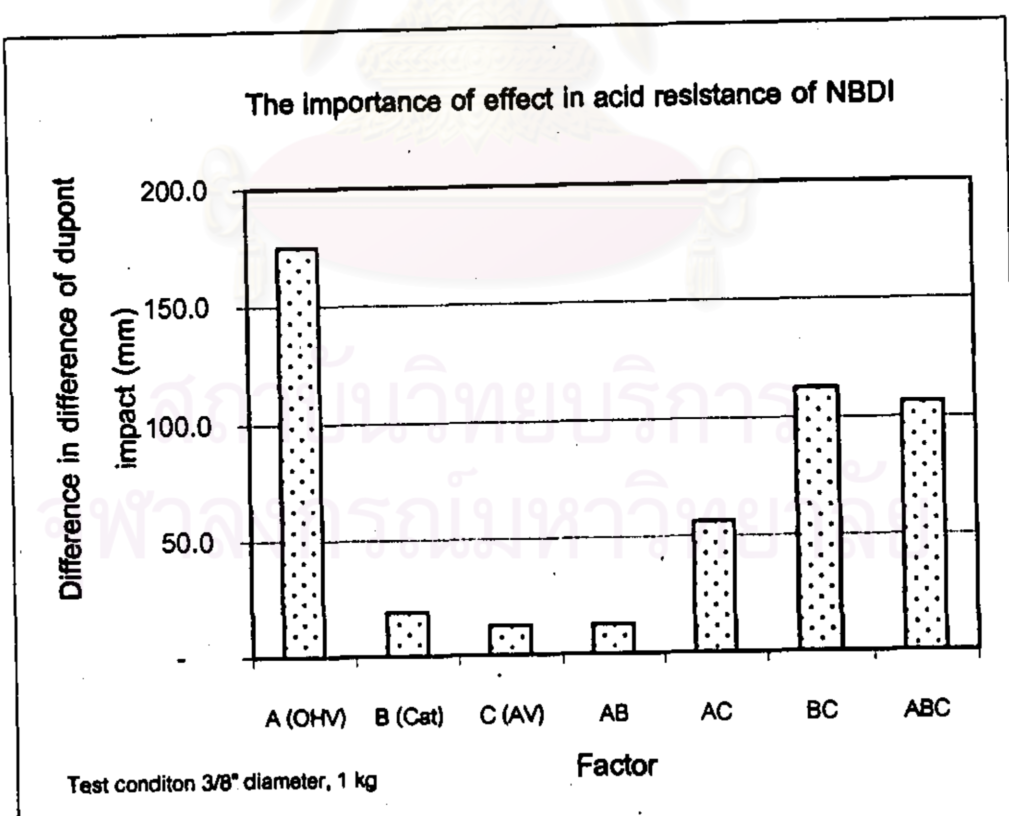

Figure 5.28 The importance of effect in acid resistance of NBDI

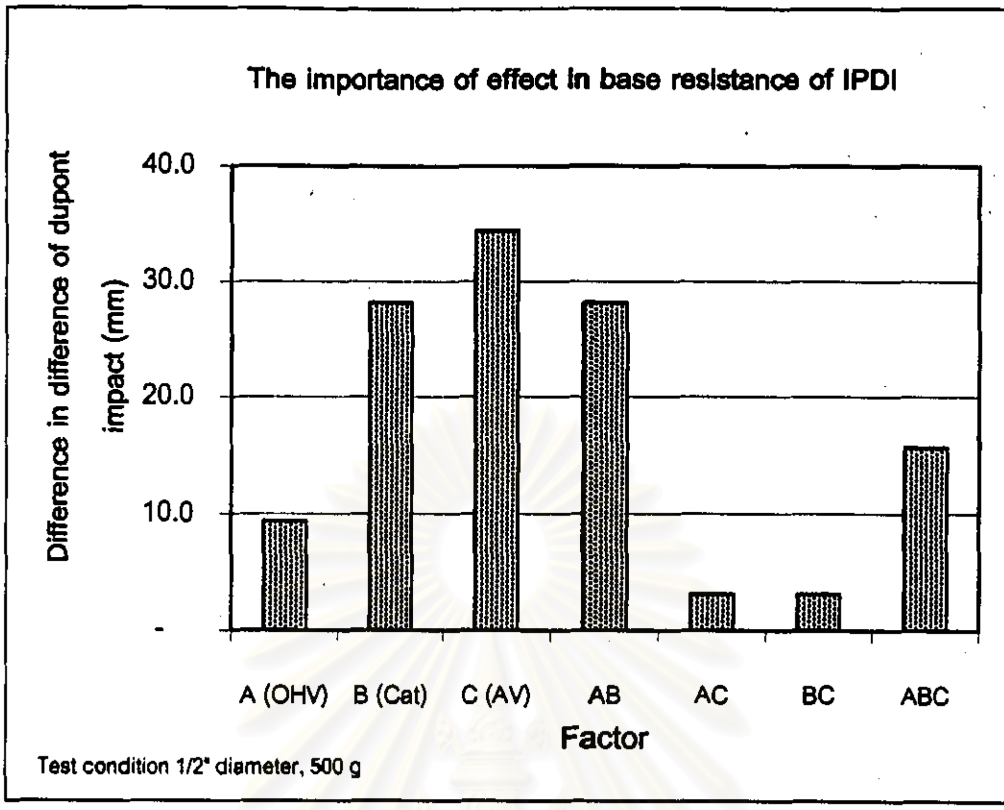

Figure 5.29 The importance of effect in base resistance of IPDI

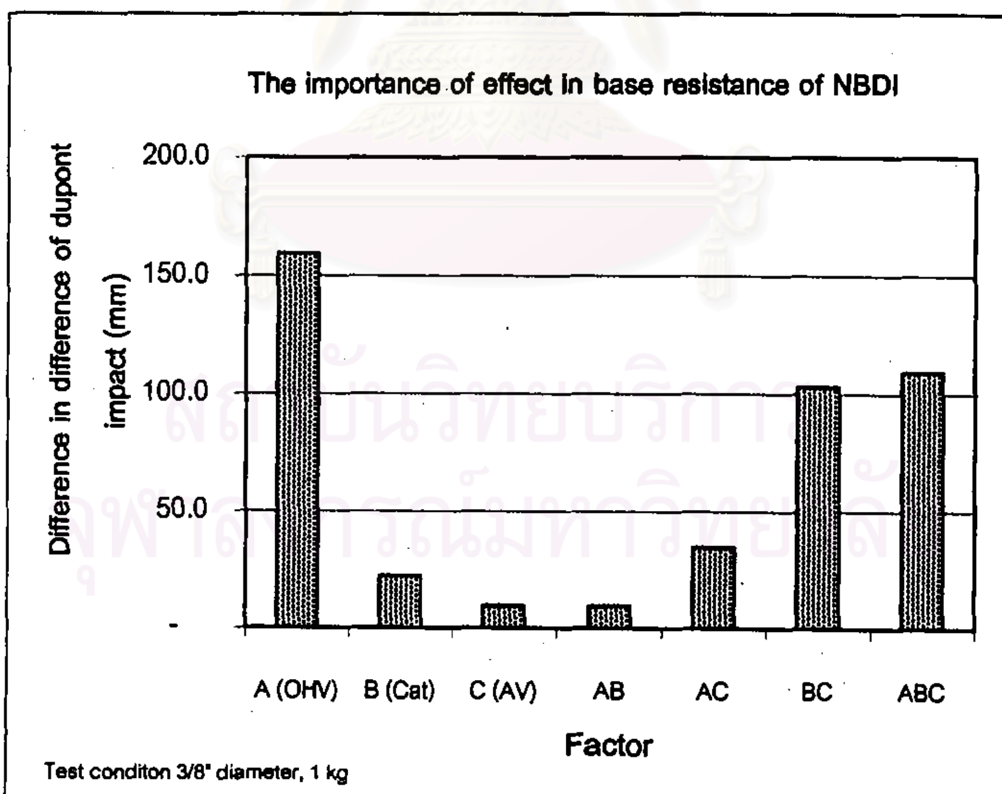

Figure 5.30 The importance of effect in base resistance of NBDI

***Table 5.5** The importance of effect in acid resistance of IPDI*

	A (OHV)	B (Cat)	C (AV)	AB	AC	BC	ABC
Avg(-)	21.9	21.9	15.6	-	12.5	12.5	15.6
Avg(+)	15.6	15.6	21.9	37.5	25.0	25.0	21.9
Δ	- 6.3	- 6.3	6.3	37.5	12.5	12.5	6.3

Note: Test condition 1/2" diameter, 500 g

***Table 5.6** The importance of effect in acid resistance of NBDI*

	A (OHV)	B (Cat)	C (AV)	AB	AC	BC	ABC
Avg(-)	209.4	112.5	128.1	128.1	93.8	65.6	175.0
Avg(+)	34.4	131.3	115.6	115.6	150.0	178.1	68.8
Δ	- 175.0	18.8	- 12.5	- 12.5	56.3	112.5	- 106.3

Note: Test condition 3/8" diameter, 1 kg

***Table 5.7** The importance of effect in base resistance of IPDI*

	A (OHV)	B (Cat)	C (AV)	AB	AC	BC	ABC
Avg(-)	34.4	43.8	12.5	43.8	28.1	31.3	21.9
Avg(+)	25.0	15.6	46.9	15.6	31.3	28.1	37.5
Δ	- 9.4	- 28.1	34.4	- 28.1	3.1	- 3.1	15.6

Note: Test condition 1/2" diameter, 500 g

***Table 5.8** The importance of effect in base resistance of NBDI*

	A (OHV)	B (Cat)	C (AV)	AB	AC	BC	ABC
Avg(-)	212.5	121.9	128.1	137.5	115.6	81.3	187.5
Avg(+)	53.1	143.8	137.5	128.1	150.0	184.4	78.1
Δ	- 159.4	21.9	9.4	- 9.4	34.4	103.1	- 109.4

Note: Test condition 3/8" diameter, 1 kg

5.Drying Time Test and Gel Time test.

Drying time is defined by three values, set to touch time (t_1), tack free time (t_2) and dry hard time (t_3). The t_1 and t_2 are values resulting from the evaporation rate of solvent. The higher evaporation rate, the lower the value of t_1 and t_2. The dry hard time (t_3), the most important value, is the time that the film is hard enough to protect the film from damage in mild condition such as rubbing strongly with finger. The dry hard time is resulted from the reaction rate of polyol and hardener. So, determining dry hard time is an indirect method for estimating the reactivity of the hardener. Consequently, the reactivity of two hardeners is compared in this work by the dry hard time (t_3).

Following the objectives of this work, to compare between two hardeners, the various experiments are tested by measuring the drying time of one polyol simultaneously with different hardeners . So, the results of this test may not be compared among the polyols at the same hardener because the test does not occur at the same time.

The set to touch time (t_1), tack free time (t_2) and dry hard time (t_3) are demonstrated in the Table 5.9. The set to touch time (t_1) and tack free time (t_2) of two hardeners are very close. The small difference may result from the preparing film step but it can not affect the performance of the film.

From Figure 5.31, the IPDI samples have the dry hard time (t_3) in the range of 29-32 minutes while the NBDI samples have the dry hard time (t_3) in the range of 19-20 minutes. All of the IPDI samples take longer time than the NBDI samples to achieve dry hard film. So it can be concluded that the NBDI samples have more reactivity than the IPDI samples.

In this work, the viscosity measurement of the coating solutions by ford cup #4 is used. Because acid value (AV) is the main factor that influences on gel time [Kano,1999], the polyols which are different in AV were selected. To study this factor in NBDI, thus, two couples of polyols (7N-8N and 9N-10N) were used in this experiment. In addition, to compare between NBDI and IPDI, two samples of IPDI (7I and 8I) were tested as well.

In the gel time test, from Figure 5.32, the similar results were obtained from both couples of samples. The higher AV, the shorter gel time in both 7N-8N and 9N-10N samples. It can be explained that the increase of AV can accelerate the reaction rate and shorten the gel time. To compare the difference of two hardeners, in figure 5.33, both couples of samples (7N-7I and 8N-8I) gave the same tendency. The coatings from NBDI has much higher reactivity than the coatings from IPDI. In table 5.9, the numerical values of viscosity at the different time are depicted. It can be seen clearly that, for example, 8N is gel within 3 hours whereas 8I is still not gel and the viscosity of 8I is only 43.4 seconds, by ford cup #4. It can be concluded from the results that AV affects on the reaction rate of polyurethane formation in this system. The influence may come from deionization of proton from carboxyl group of resin. The proton which acts as a catalyst takes part in the mechanism of reaction and increases reaction rate. [Nakamichi and Ishidoya, 1988] Apart from AV, the type of hardener also plays a vital role in the reaction rate. The reason that NBDI gives shorter gel time is still unobvious. It can be simply explained that the difference in gel time derives from the reactivity of isocyanate group in NBDI higher than that of isocyanate group in IPDI.

Additionally, it can be found that the influence of AV and the type of hardeners on the gel time is in the same trend as the influence of that on drying time.

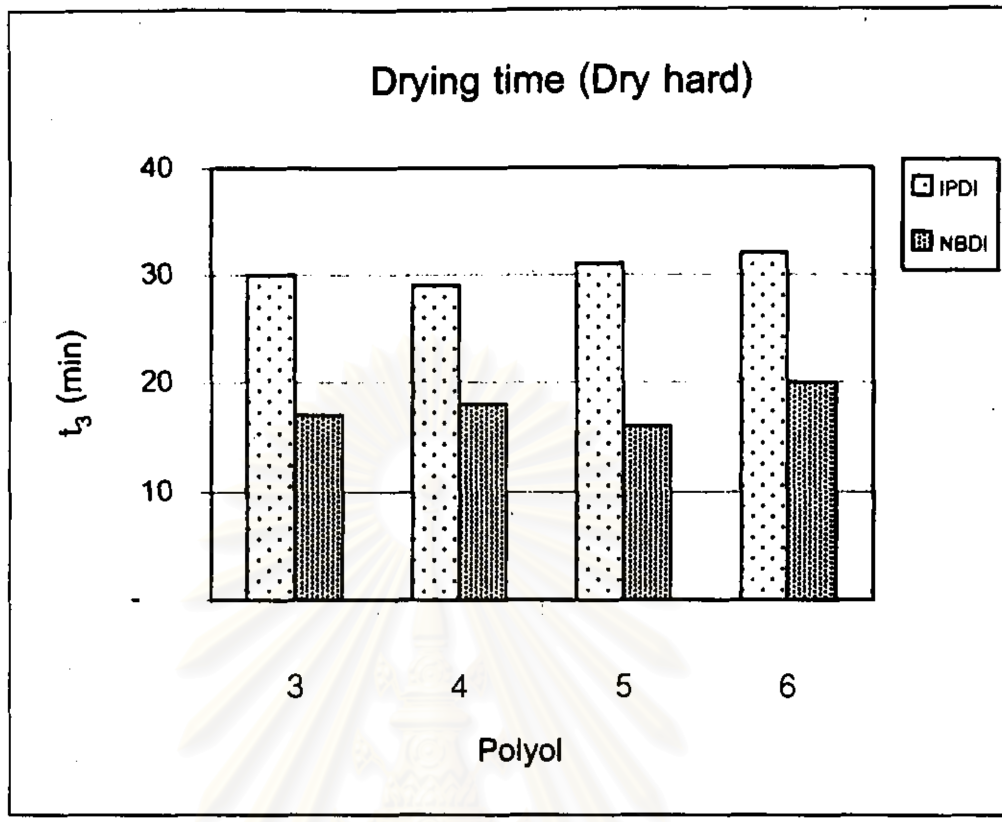

Figure 5.31 Dry hard time (t_3)

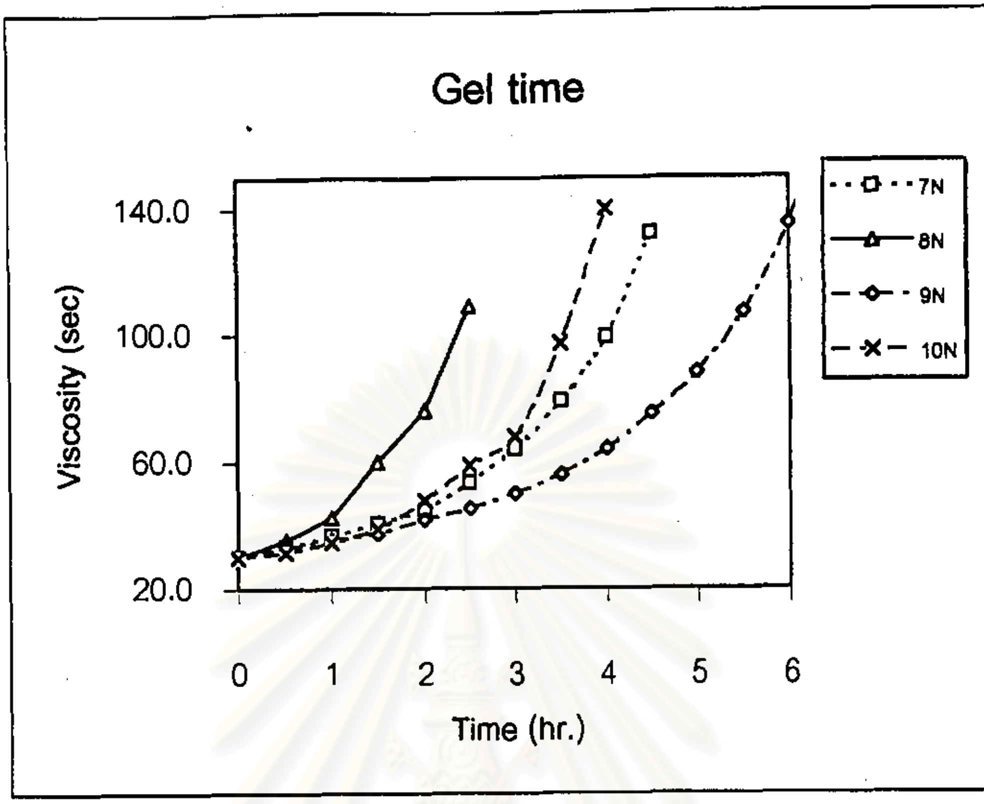

Figure 5.32 Gel time of NBDI sample

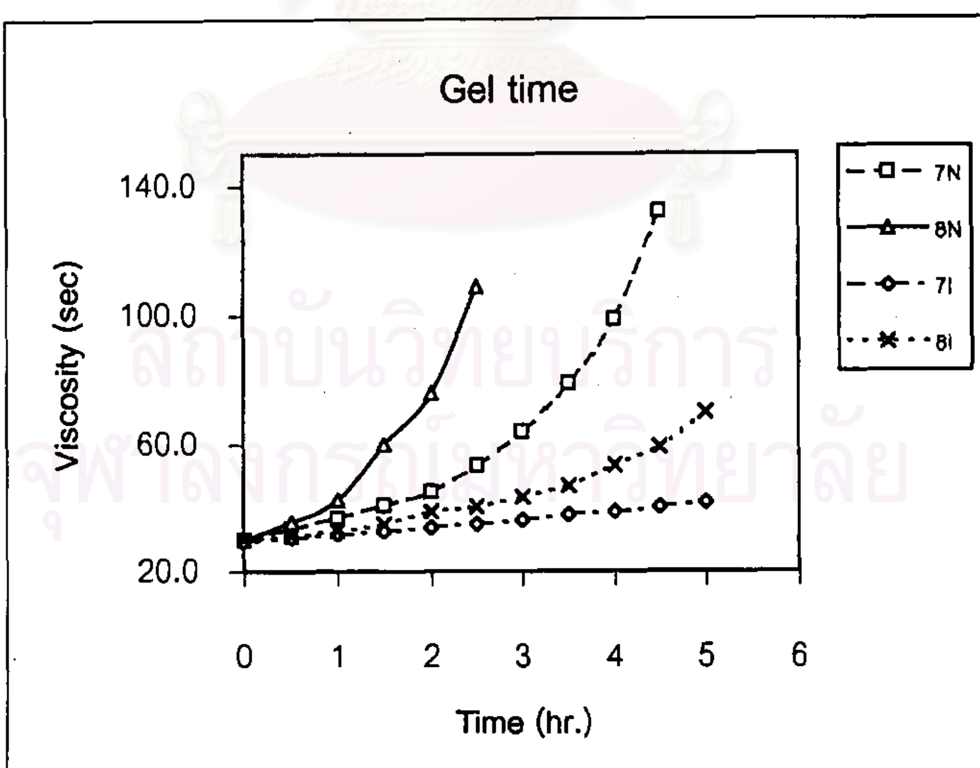

Figure 5.33 Gel time comparison between NBDI and IPDI

Table 5.9 Drying time

Sample	t_1 (min)	t_2 (min)	t_3 (min)	Sample	t_1 (min)	t_3 (min)	t_3 (min)
3I	4' 40"	6' 00"	30'	*3N*	3' 40"	5' 00"	17'
4I	4' 20"	5' 00"	29'	*4N*	3' 40"	5' 00"	18'
5I	4' 45	5' 20"	31'	*5N*	4' 10"	4' 50"	16'
6I	4' 15	5' 20"	32'	*6N*	4' 00"	4' 40"	20'

Table 5.10 Gel time test

Time	Viscosity by ford cup #4 (sec.)					
(hr.)	7N	8N	9N	10N	7I[2]	8I
0	30.0	30.0	30.0	30.0	29.4	30.0
0.5	33.4	35.6	33.0	31.5	30.6	31.3
1.0	36.7	42.5	35.3	34.8	31.9	32.9
1.5	40.8	60.0	37.5	39.0	32.6	34.9
2.0	45.2	76.0	41.9	47.9	33.8	38.8
2.5	53.5	109.0	45.4	59.0	35.0	40.4
3.0	64.0	*Gel*	50.0	68.0	36.1	43.4
3.5	79.0		56.1	97.0	37.8	46.9
4.0	99.0		64.0	140.0	38.8	53.5
4.5	132.0		75.0	*Gel*	40.2	59.1
5.0	*Gel*		88.0		41.6	70.0
5.5			107.0			
6.0			135.0			
6.5			179.0			
7.0			*Gel*			

[2] The sample 7I and 8I is still not gel.

6.Weathering test

6.1 Exposure Test.

Exposure test is one of the methods to find the weatherability of coating film. The test panels are brought to the outdoor condition then the gloss of film is measured every two weeks in this work in order to observe the change of gloss.

It is more practical to change the gloss into the gloss retention in order to study the durability of the films. The gloss retention is the ratio of the gloss in that time divided by the initial gloss.

To compare the weatherability of coating film prepared by two different hardeners, the gloss retention of samples is presented in Figure 5.34. The gloss retention of NBDI samples reduces less than that of IPDI samples so it means that the weatherability of NBDI samples is better that of IPDI samples.

6.2 QUV test.[3]

In QUV test, the testing panels are exposed in the apparatus which simulates the weathering condition. In Figure 5.35, the gloss retention of both NBDI and IPDI samples are not less than 100% throughout the testing period, 700 hours. The yellowness of NBDI as shown in Figure 5.36 is more than that of IPDI but the difference is not so significant.

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[3] The experimets were conducted at MCI's laboratory, Japan.

The gloss retention of QUV test, however, is quite different from that of exposure test. QUV test is generally more severe condition than the exposure test because QUV test is one type of accelerated weathering devices. In contrast, the results of exposure test were worse than that of QUV test. This may result from the harsh location of exposure test. In this work, the panels are subjected to the uppermost stair of TMSC's[4] building. The panels may be exposed to organic vapor which comes from hood duct. The organic vapor may affect directly on the gloss of films in the long run.

[4] TMSC is Thai Mitsui Specialty Chemicals, Co.

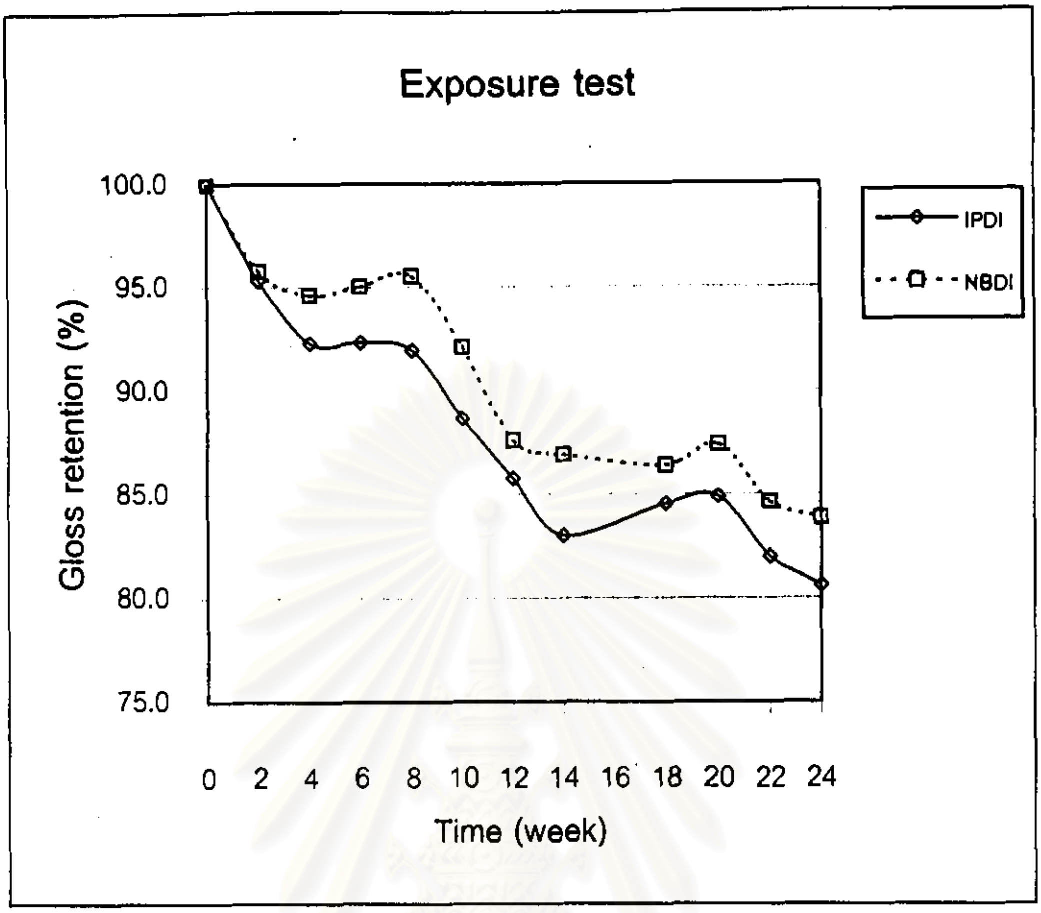

Figure 5.34 Exposure test of two hardeners

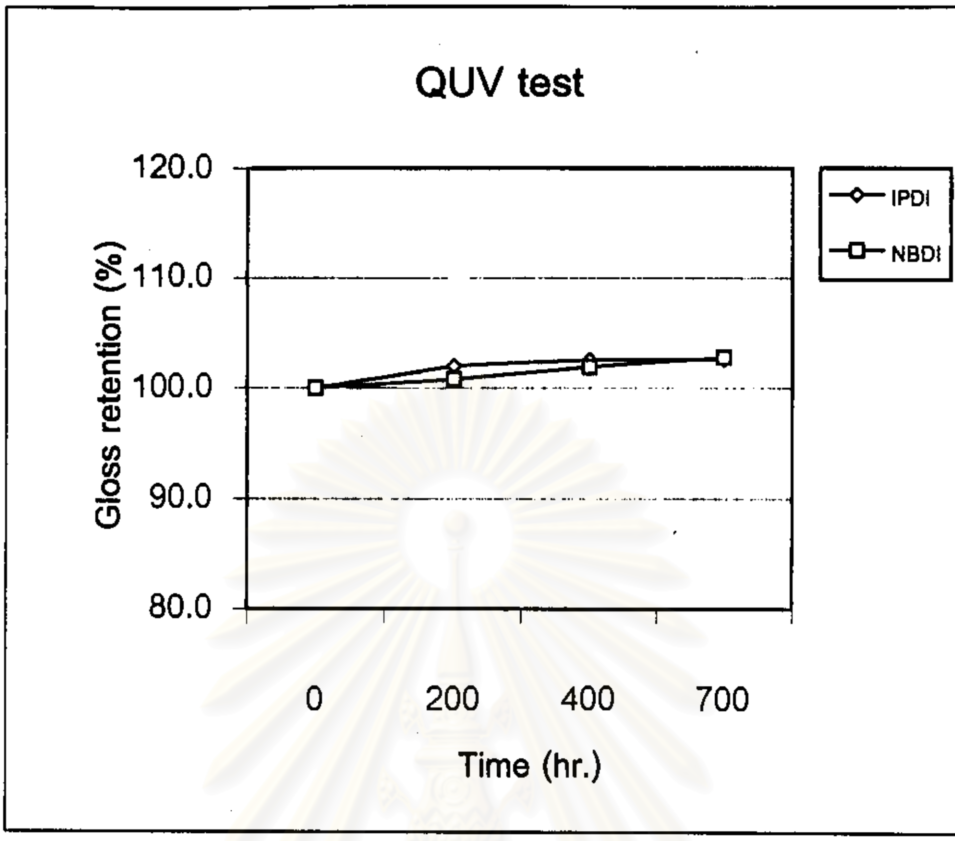

Figure 5.35 Gloss retention of QUV test

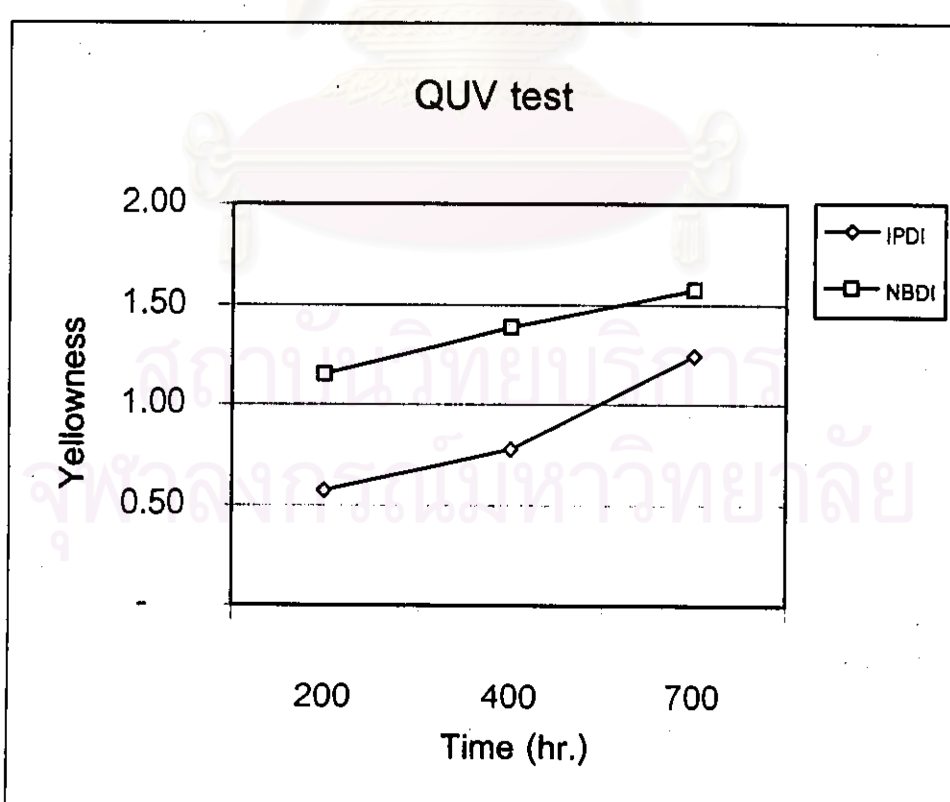

Figure 5.36 Yellowness of QUV test

7.Tensile Property Test.

The calculated tensile strength at yield and elongation at break were studied. Comparing the two hardeners, in Figure 5.37 and 5.38 the strength of IPDI samples appears to be between 300-380 kg/cm^2 while NBDI samples have the strength between 220-250 kg/cm^2. Furthermore, IPDI samples have elongation at break at 4.5-7% while NBDI samples have elongation at break 32-48%. Consequently, it can be concluded that IPDI has more tensile strength than NBDI but NBDI has more elongation at break than IPDI.

Studying the factors from polyols, only two factors, OHV and AV, are studied. So four polyols, number 5, 6, 9 and 10, are selected. Concerning the OHV factor, in both IPDI and NBDI, the yield strength increased with increasing OHV as shown in Figure 5.39. In contrast, as seen in Figure 5.40, the elongation at break of NBDI increases while IPDI decreases with increasing OHV. The yield strength of coating films increases when the OHV is increased because of the more crosslinked density film.

About the AV factor, in both IPDI and NBDI, the yield strength increases with increasing AV as presented in Figure 5.41 but the influence of AV on the yield strength is much less than OHV. In addition, the elongation at break of NBDI decreases while that of IPDI slightly increases with increasing AV.

Based on the experimental design concept, the importance of each factor presented in Figure 5.43, 5.44, 5.45 and 5.46, and the numerical value of it is shown in table 5.10-5.13. In Figure 5.43 and 5.44, the yield strength of both IPDI and NBDI is mainly impacted by OHV while the other factors produce little effects. For elongation at break, the significance of factors is demonstrated in Figure 5.45 and

5.46. The most important effect is still to be OHV. Moreover, similar to yield strength, the other factors give small effects too.

In Figure 5.47, the stress and strain of sample 5N and 5I are plotted. The area under curve represents to the toughness of the material. It is very obvious that NBDI has much more toughness than IPDI.

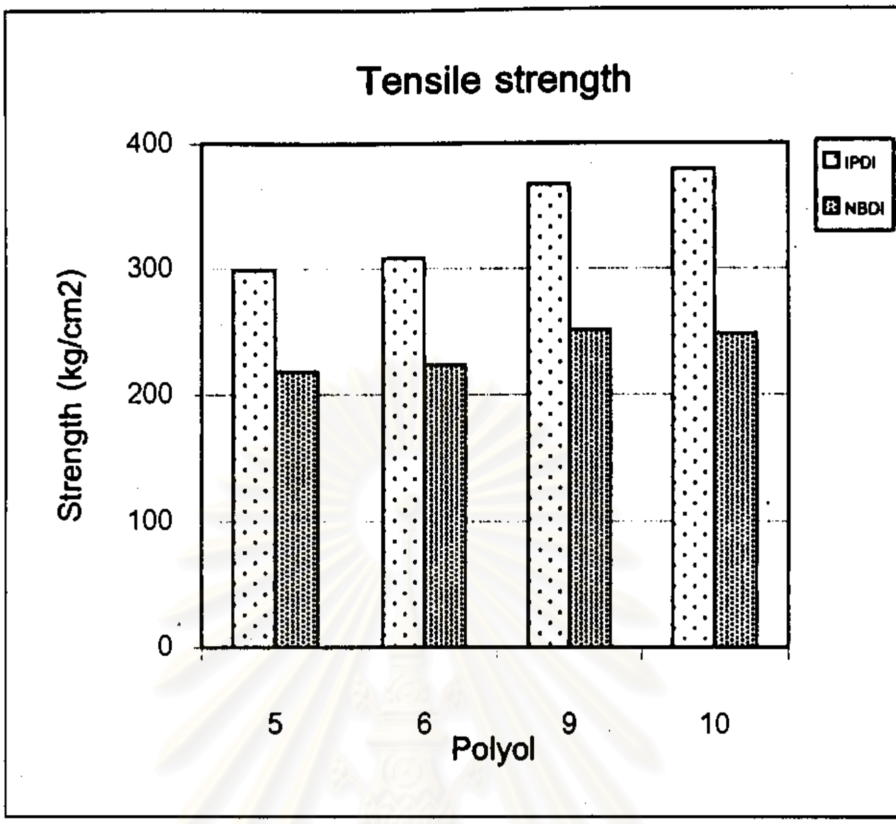

Figure 5.37 Tensile strength at yield

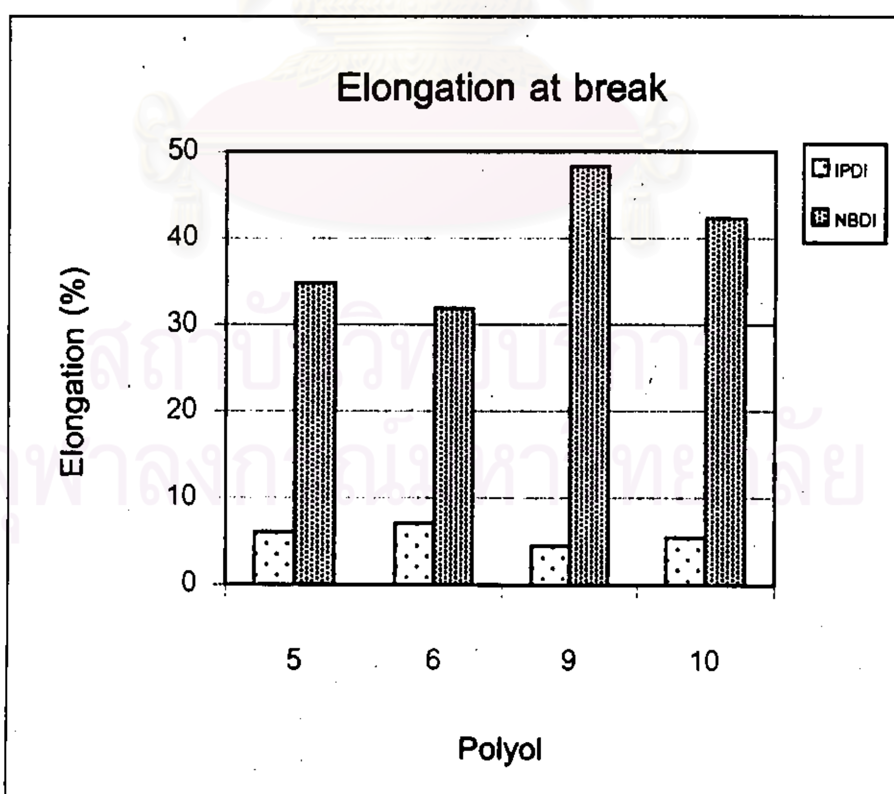

Figure 5.38 Elongation at break

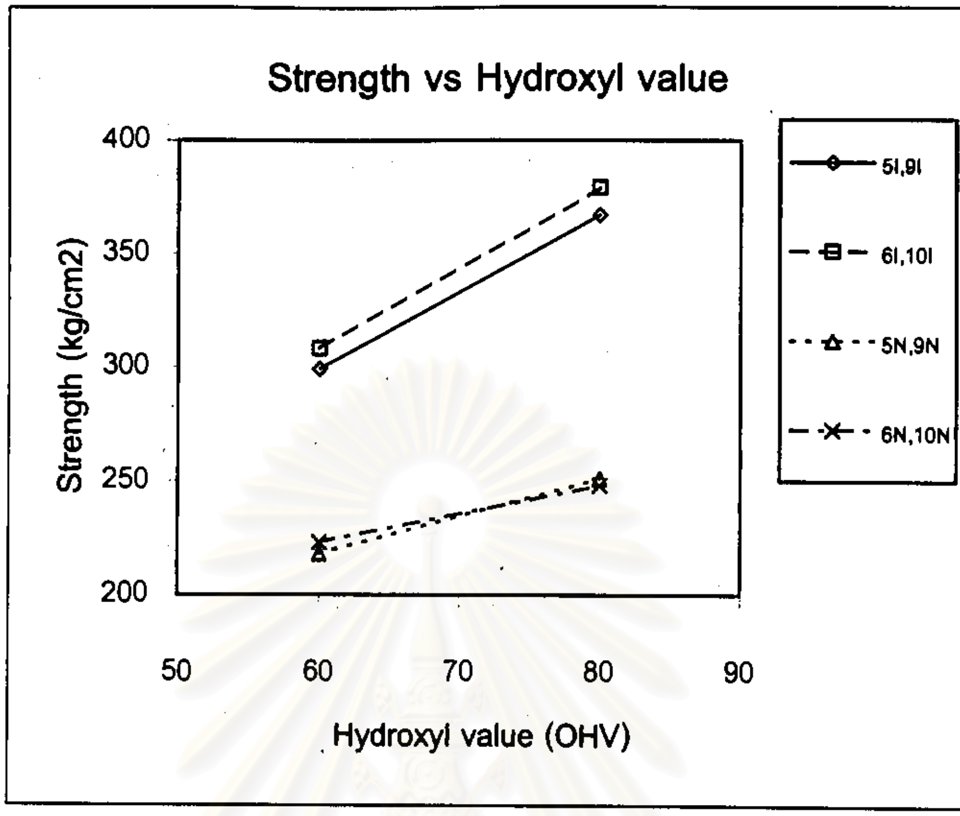

Figure 5.39 Tensile strength at yield vs hydroxyl value

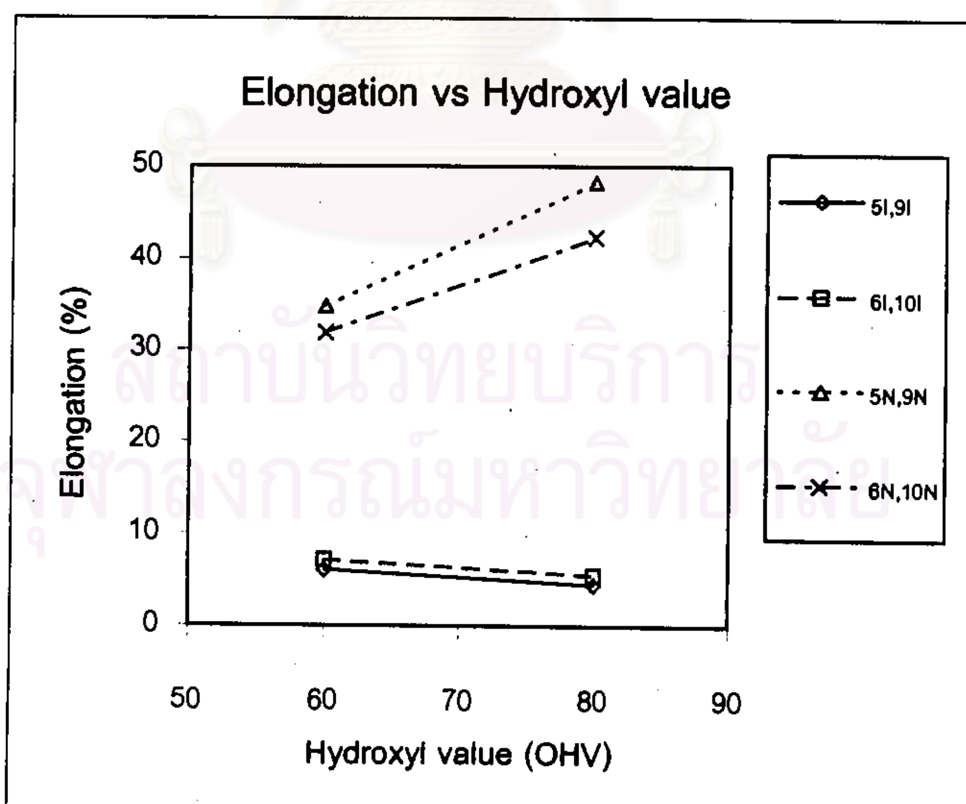

Figure 5.40 Elongation at break vs hydroxyl value

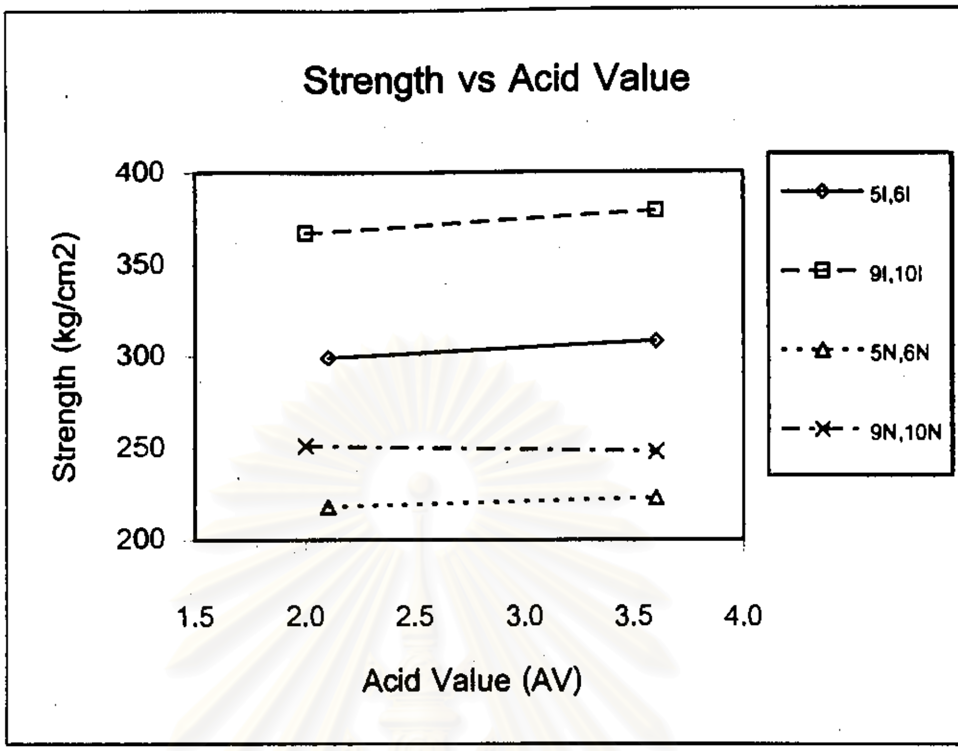

Figure 5.41 Tensile strength at yield vs acid value

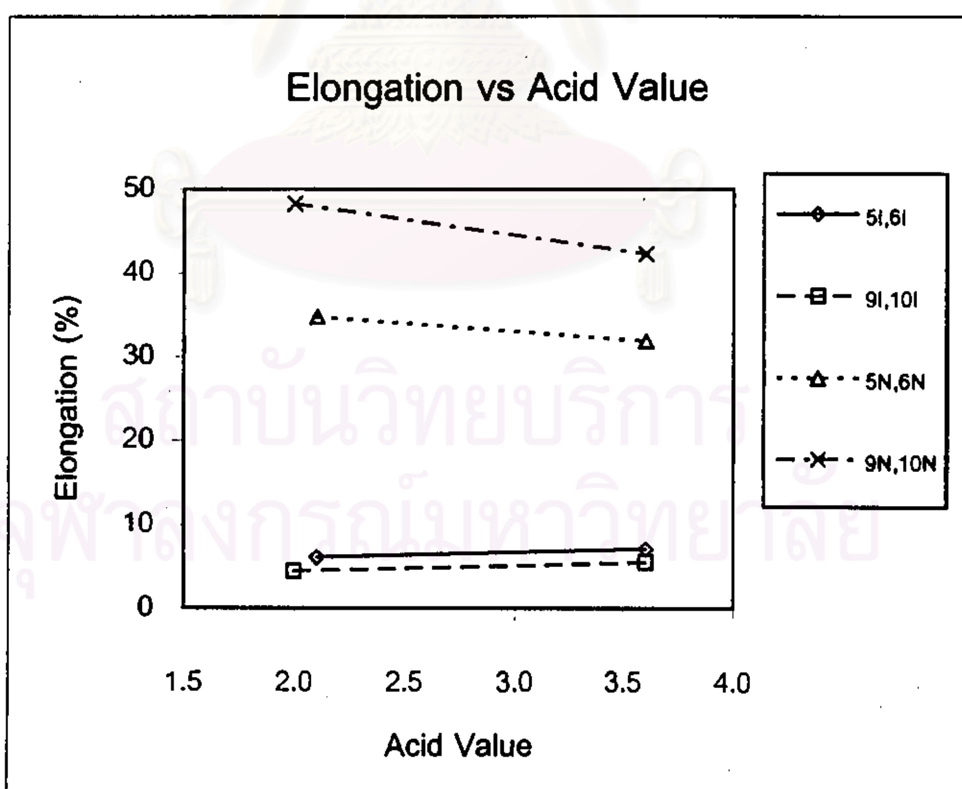

Figure 5.42 Elongation at break vs acid value

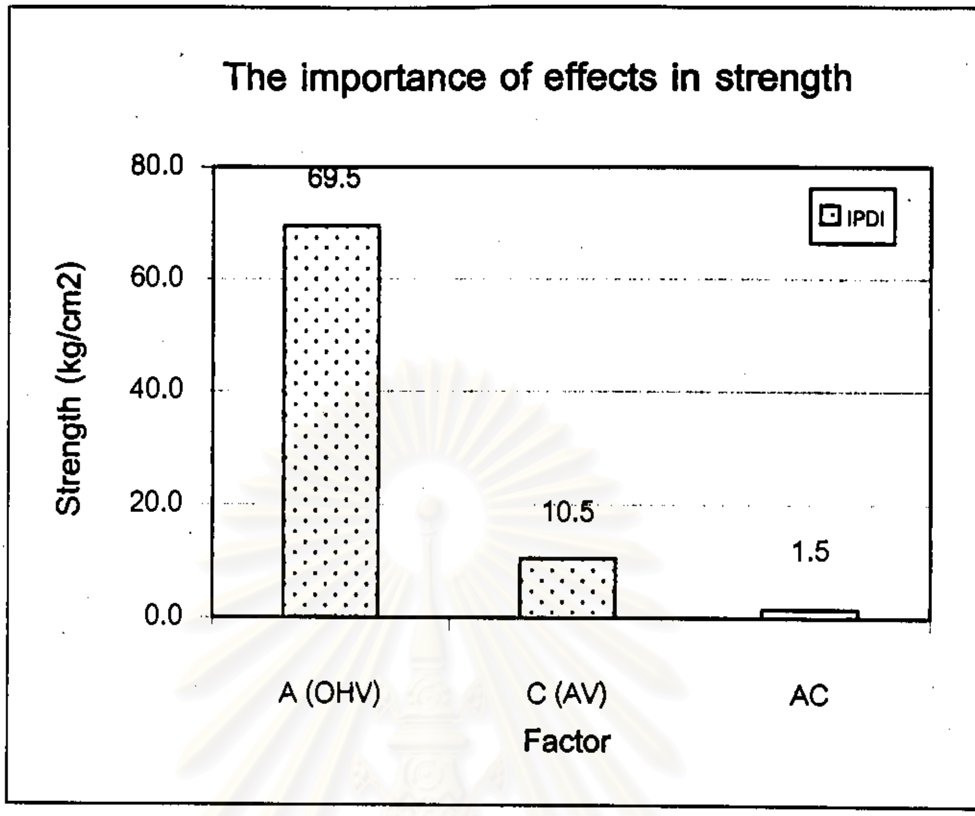

Figure 5.43 The importance of effects in yield strength of IPDI

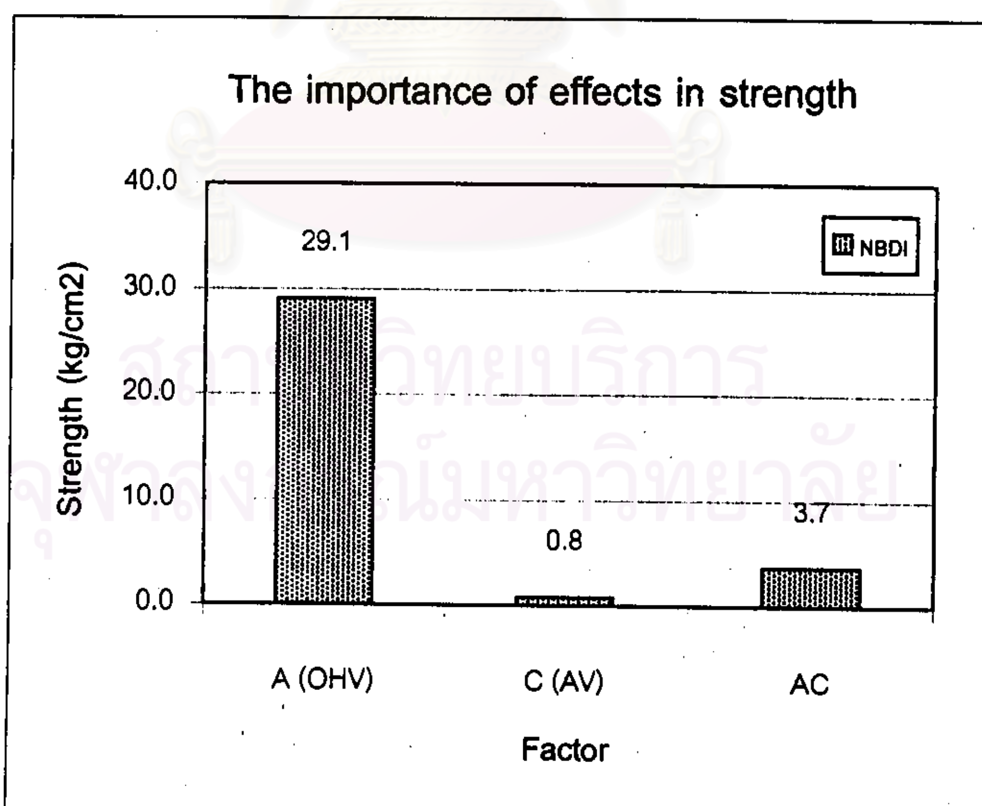

Figure 5.44 The importance of effects in yield strength of NBDI

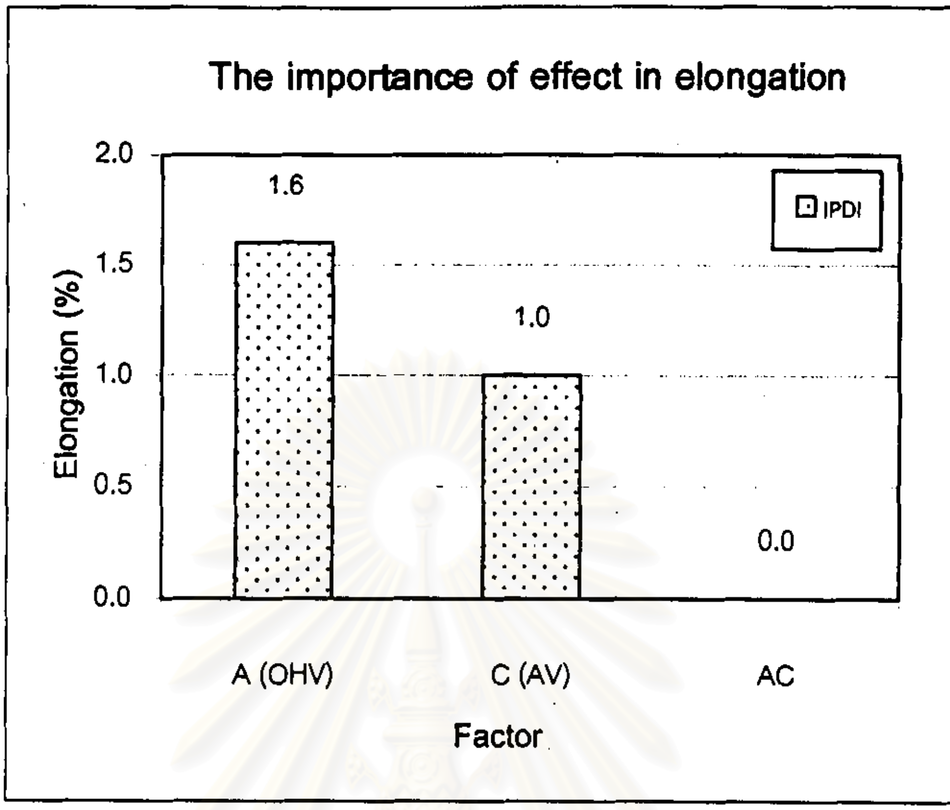

Figure 5.45 The importance of effects in elongation at break of IPDI

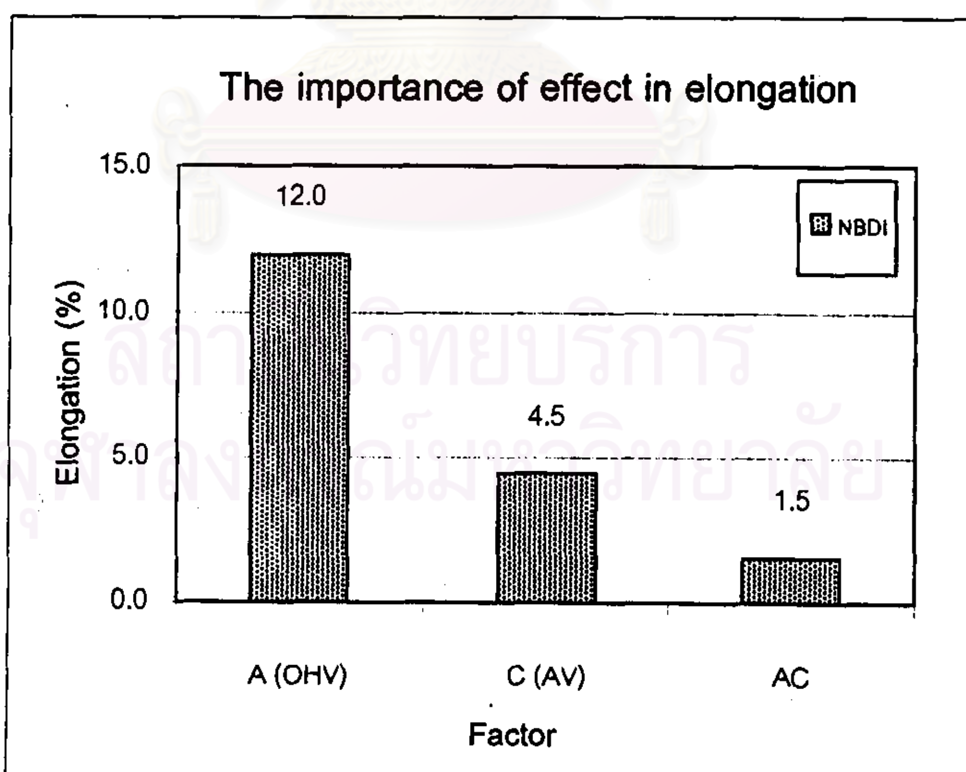

Figure 5.46 The importance of effects in elongation at break of NBDI

***Table 5.11** The importance of effect in yield strength of IPDI*

Effect	A (OHV)	C (AV)	AC
Avg(-)	303.7	333.2	337.6
Avg(+)	373.1	343.6	339.1
Δ	69.5	10.5	1.5
MSB_{effect}	28,964.1	658.9	13.6
F_{effect}	34.0	0.8	0.0

F_C (0.95,1,24) = 4.26 or F_C (0.99,1,24) = 7.82

***Table 5.12** The importance of effect in yield strength of NBDI*

Effect	A (OHV)	C (AV)	AC
Avg(-)	220.6	234.8	237.0
Avg(+)	249.7	235.5	233.3
Δ	29.1	0.8	- 3.7
MSB_{effect}	4,443.2	3.0	70.9
F_{effect}	17.3	0.0	0.3

F_C (0.95,1,21) = 4.33 or F_C (0.99,1,20) = 8.03

***Table 5.13** The importance of effect in elongation at break of IPDI*

Effect	A (OHV)	C (AV)	AC
Avg(-)	6.6	5.3	5.8
Avg(+)	5.0	6.3	5.8
Δ	-1.6	1.0	0.0
MSB_{effect}	15.9	5.7	0.0
F_{effect}	5.3	1.9	0.0

F_C (0.95,1,24) = 4.26

***Table 5.14** The importance of effect in elongation at break of NBDI*

Effect	A (OHV)	C (AV)	AC
Avg(-)	33.3	41.6	40.1
Avg(+)	45.3	37.1	38.6
Δ	12.0	-4.5	-1.5
MSB_{effect}	753.6	104.4	12.4
F_{effect}	5.2	0.7	0.1

F_C (0.95,1,21) = 4.33

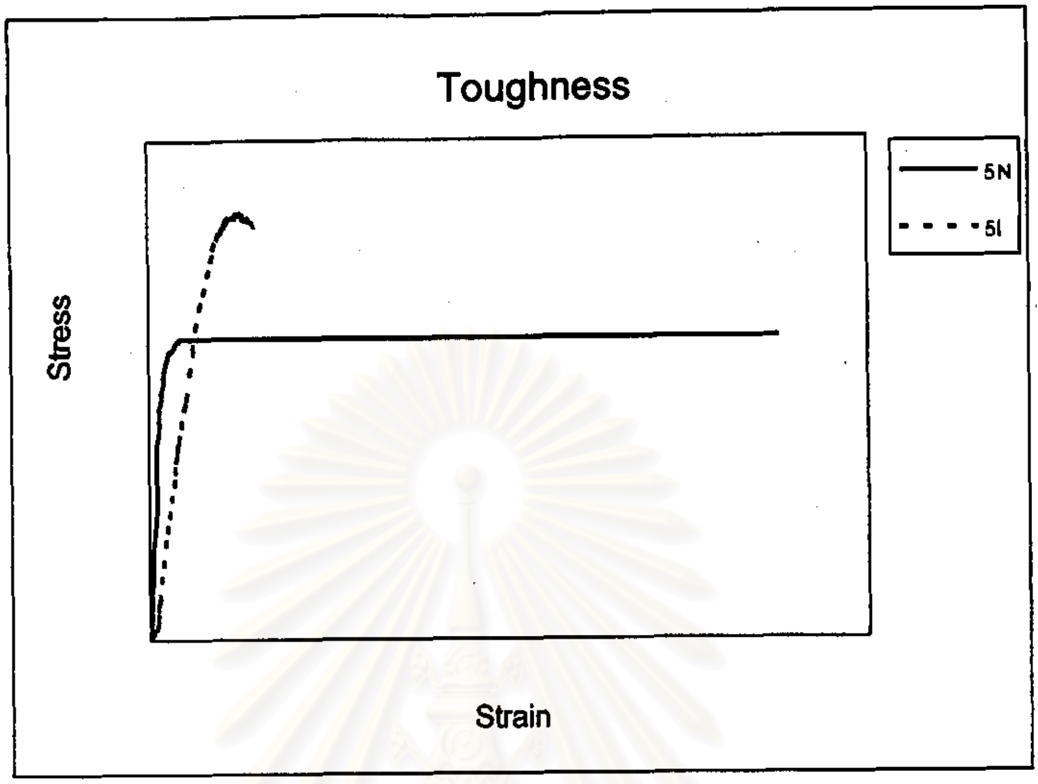

Figure 5.47 Stress-Strain curve of 5N and 5I

8. Dynamic Mechanical Analysis (DMA) test

Dynamic mechanical test is one of the useful methods to determine the behavior of material. From the DMA results, the glass transition temperature (Tg) is generally the point at the maximum of loss tangent (tan δ). In addition, the crosslinking density (XLD) of film are calculated form DMA results. So, the Tg and crosslinking density (XLD) of samples are demonstrated in table 5.14 and the importance of three factors is presented in table 5.15. It can be seen clearly that the most significant factor that affects on Tg is OHV. The higher the OHV , the higher the Tg. In contrast, increase of the quantity of catalyst, lowering MW, results in decreasing of the Tg. Concerning AV, the trend is similar to OHV but the degree of changing in Tg is the least. Known from the basic concept of polymer properties, increase of OHV will increase XLD and lead to higher Tg. The influence of AV, however, is still ambiguous. The AV affects directly on the extent of reaction which might affect on the network formation during curing. [Nakamichi and Ishidoya, 1988] As a result, some of isocyanate group may react with moisture in the air instead of hydroxyl group in polyol. Hence, the structure of molecule may be affected by this reason.

Comparing between two hardeners, all of NBDI films produce lower Tg, approximately 10°C than IPDI films. So NBDI gives softer film, and has more toughness than IPDI films as shown in Figure 5.47. Similarly, the XLD of NBDI films are also lower than that of IPDI films by about 16 %. Consequently, it can be summarized that NBDI film gives higher dupont impact because of lower Tg but it gives lower chemical resistance due to lower XLD [Kano, 1999].

Table 5.15 Glass transition temperature (Tg) and Crosslinking density (XLD) of film[5]

Sample	Tg (°C)	XLD[6] (mol/cc)	Sample	Tg (°C)	XLD (mol/cc)
3N	65.4	1.71E-02	***3I***	-	-
4N	69.8	1.79E-02	***4I***	-	-
5N	64.6	2.00E-02	***5I***	78.3	1.88E-02
6N	65.3	1.67E-02	***6I***	74.7	2.01E-02
7N	79.0	1.54E-02	***7I***	-	-
8N	-	-	***8I***	-	-
9N	69.9	1.61E-02	***9I***	80.2	2.04E-02
10N	74.8	1.63E-02	***10I***	76.0	2.00E-02

Table 5.16 The importance of effect in Tg

Effect	A (OHV)	B (Cat)	C (AV)
Avg (-)	65.1	71.4	66.6
Avg (+)	74.6	66.6	70.0
Δ	9.5	-4.8	3.4

[5] The results were supplied by MCTs laboratory, Japan

[6] The XLD calculation can be seen in appendix C.4