

**CHAPTER IV**  
**RESULTS & DISCUSSION**

**4.1 Mechanical Testing**

Two different kinds of vulcanized rubber films were determined their physical properties and results are shown in Table 4.1.

**Table 4.1** Results for the testing of NRL and DPNR vulcanized rubber films

Types of Latex	Sample	Unaging Tensile Strength(MPa)	Aging Tensile Strength(MPa)	Unaging Tear Strength(N/mm)	Aging Tear Strength(N/mm)	Hardness
NRL	DRD1	15.01 ± 2.71	14.24 ± 2.13	39.87 ± 2.53	32.85 ± 2.74	25.5
	DRD3	14.36 ± 2.62	13.64 ± 1.78	38.12 ± 2.30	37.9 ± 2.91	24
	DRD5	13.42 ± 0.89	12.11 ± 2.04	33.79 ± 2.88	33.26 ± 6.40	25
	DRD7	11.2 ± 1.93	10.8 ± 1.77	31.08 ± 3.24	20.45 ± 4.51	27.5
	DRD9	19.94 ± 2.45	19.12 ± 3.33	54.04 ± 2.95	51.54 ± 3.84	26.5
	DRD11	20.37 ± 4.27	18.86 ± 1.33	56.56 ± 2.60	50.03 ± 2.87	28.5
	DRD13	15.11 ± 2.53	14.51 ± 1.05	43.71 ± 2.61	41.35 ± 1.91	29.5
	DRD15	14.54 ± 2.86	14.41 ± 1.25	41.29 ± 3.06	41.18 ± 2.69	30
	DRD17	19.99 ± 1.11	18.34 ± 4.09	54 ± 4.43	52.39 ± 4.52	28
	DRD19	20.44 ± 1.35	20.34 ± 1.32	55.19 ± 3.41	53.57 ± 4.32	28.5
	DRD21	17.1 ± 1.23	16.91 ± 0.72	53.59 ± 3.30	52.87 ± 4.62	31
	DRD23	17.13 ± 0.96	16.65 ± 1.93	49.66 ± 3.73	49.41 ± 2.20	38
	DPNR	DRD2	14.26 ± 2.79	13.6 ± 1.54	35.45 ± 2.57	33.2 ± 2.71
DRD4		12.16 ± 0.91	11.23 ± 1.72	37.16 ± 2.86	34.66 ± 3.51	22
DRD6		12.15 ± 0.8	11.28 ± 1.16	33.14 ± 2.90	30.05 ± 2.95	22.5
DRD8		9.47 ± 1.49	8.57 ± 1.63	13.43 ± 1.37	8.14 ± 1.66	26
DRD10		16.05 ± 2.93	13.73 ± 2.51	46.85 ± 3.47	42.97 ± 2.33	27
DRD12		16.92 ± 2.55	14.06 ± 1.75	54.03 ± 2.08	49.49 ± 4.22	28
DRD14		12.14 ± 2.76	11.04 ± 1.65	44.93 ± 3.09	40.47 ± 2.7	28.5
DRD16		NA*	NA*	NA*	NA*	NA*
DRD18		17.26 ± 1.35	15.24 ± 2.18	50.39 ± 3.13	48.12 ± 3.15	26.5
DRD20		18.13 ± 1.98	17.95 ± 2.04	50.58 ± 4.00	50.55 ± 1.61	27
DRD22		15.64 ± 1.88	15.45 ± 1.72	50.36 ± 4.90	47.38 ± 4.33	28.5
DRD24	16.43 ± 1.88	15.63 ± 1.42	39.89 ± 4.18	37.67 ± 4.72	31.5	

NA\* = not available (cannot form film)

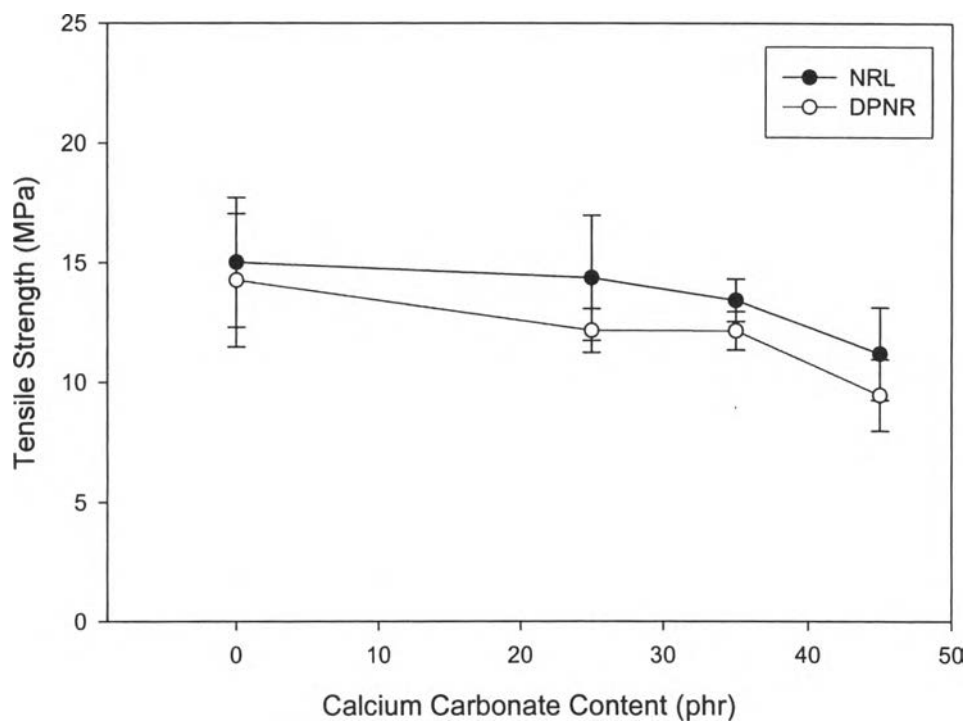
#### 4.1.1 Mechanical Properties

##### 4.1.1.1 *Commercial 3M® dental rubber dam*

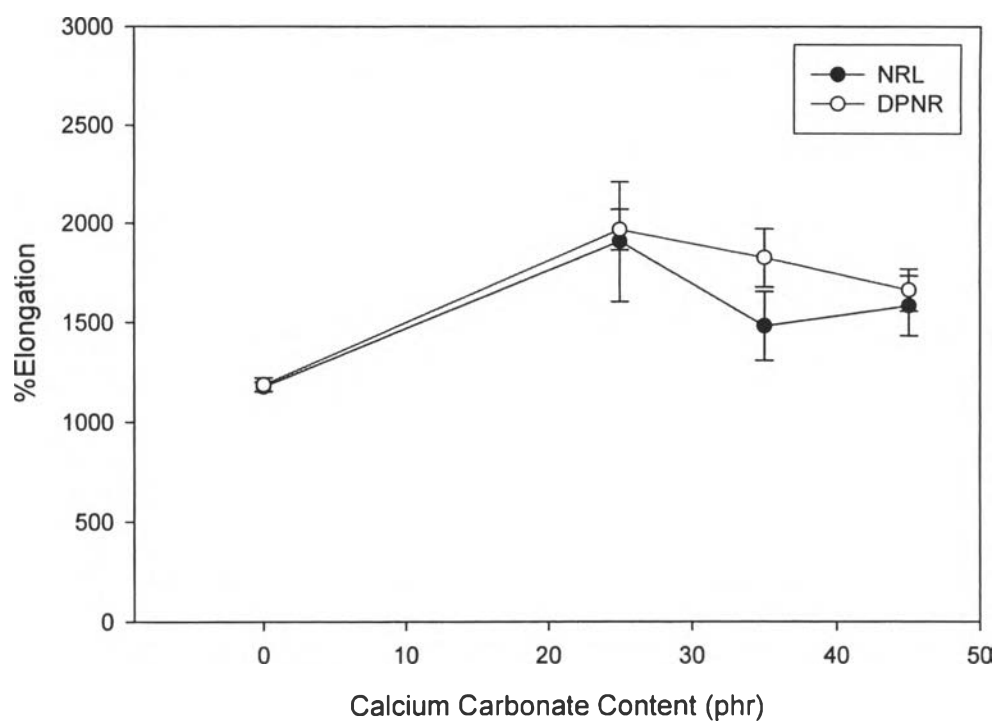
Mechanical properties of the 3M® dental rubber dam were determined and the result of the average tensile strength, tear strength, and hardness were  $20 \pm 1.16$  MPa,  $50 \pm 3.28$  N/mm, and 26 degree of hardness respectively.

##### 4.1.1.2 *Calcium carbonate*

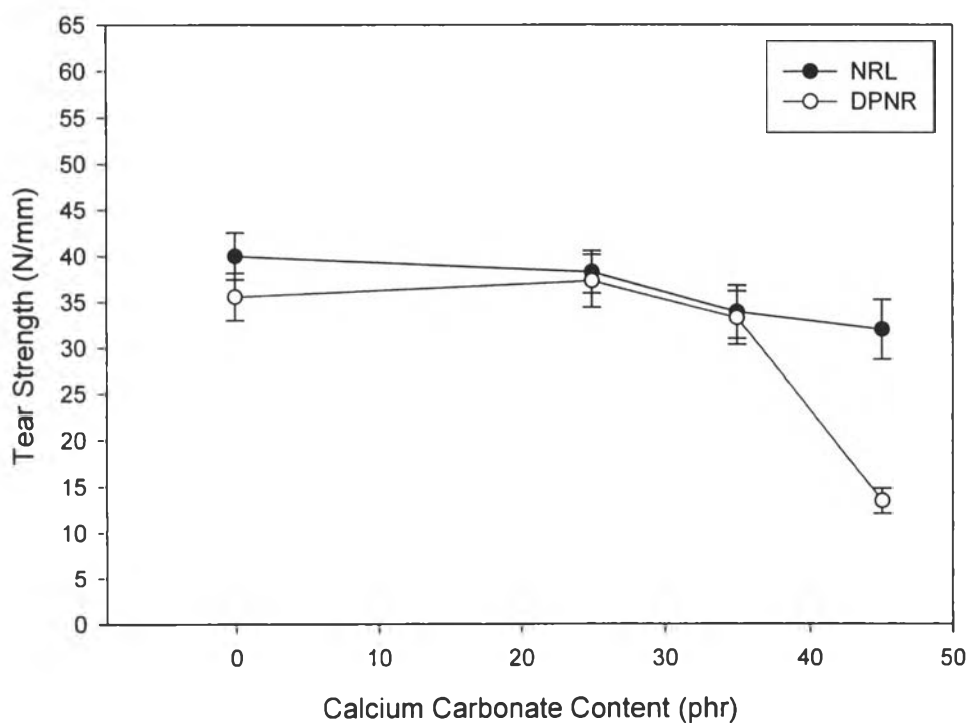
Tensile strength, elongation at break, tear strength and hardness of both vulcanized NRL and DPNR film were determined as shown in Figures 4.1, 4.2, 4.3 and 4.4 respectively.



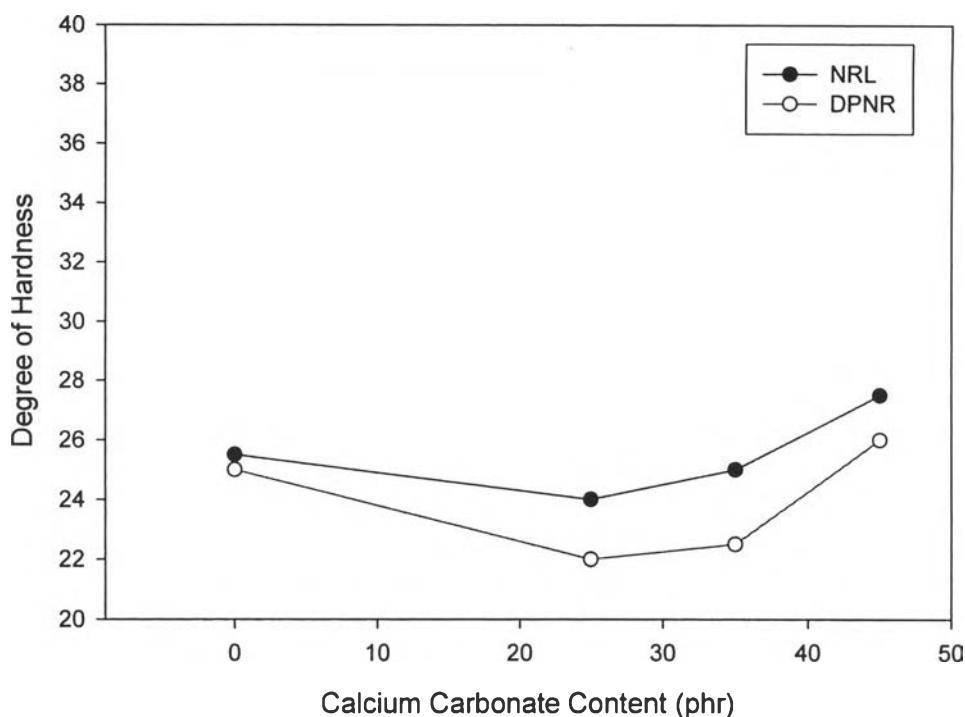
**Figure 4.1** Tensile strength of unaged vulcanized NRL and DPNR films with 50% calcium carbonate.



**Figure 4.2** %Elongation of unaged vulcanized NRL and DPNR films with 50% calcium carbonate.



**Figure 4.3** Tear strength of unaged vulcanized NRL and DPNR films with 50% calcium carbonate.



**Figure 4.4** Hardness of unaged vulcanized NRL and DPNR films with 50% calcium carbonate system.

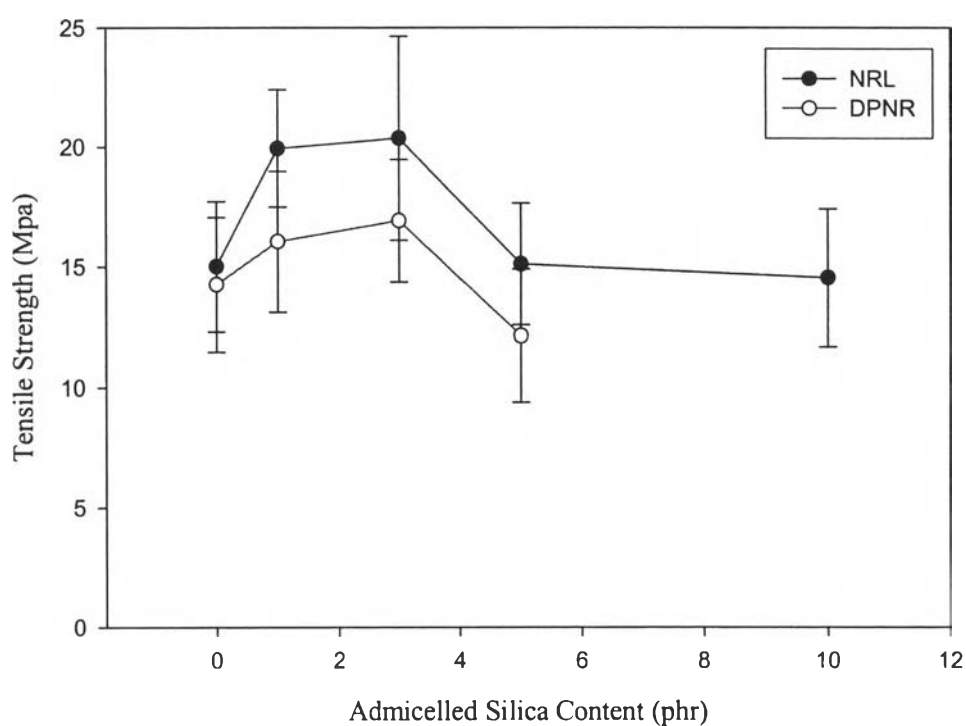
$\text{CaCO}_3$  is light inactive filler. It used to reduce latex quantity for saving cost. In crosslinking reaction,  $\text{CaCO}_3$  will disperse into rubber chain without bonding with rubber particles. This reason slightly effect on the mechanical properties of rubber film. However, if increases  $\text{CaCO}_3$  content more than 35 phr. It will strongly effect on the mechanical properties of rubber film

The results showed that the values of tensile strength, tear strength and hardness slightly decreased due to increasing of calcium carbonate content in range of 0, 25 and 35 phr. The values of tensile strength and tear strength rapidly decreased when increasing the content is more than 35 phr because of the rubber films become stiffer and the elasticity decreases from increasing of the hardness value.

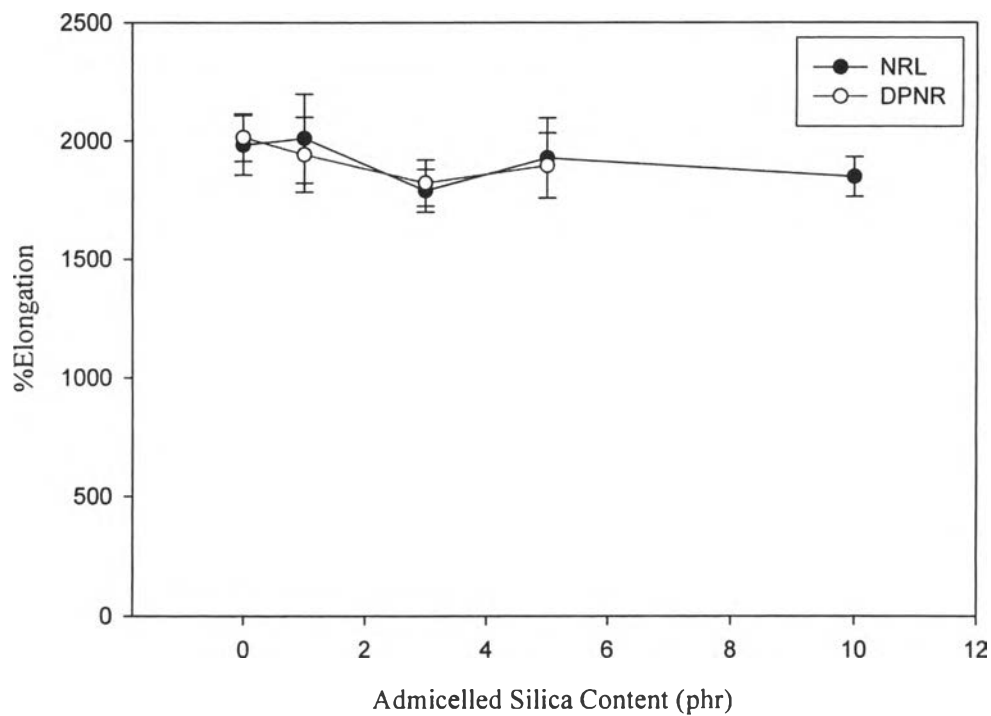
For the DPNR film, surfactant treatment is an effective means of solubilizing particle-bound proteins, but at the cost of reduced vulcanization efficiency. With a conventional cure recipe, the ultimate state of cure attained with DPNR is substantially lower than that attainable with commercial NRL (Schloman, 2002). Thus, the mechanical properties of DPNR were less than NRL.

#### 4.1.1.3 Admicelled Silica

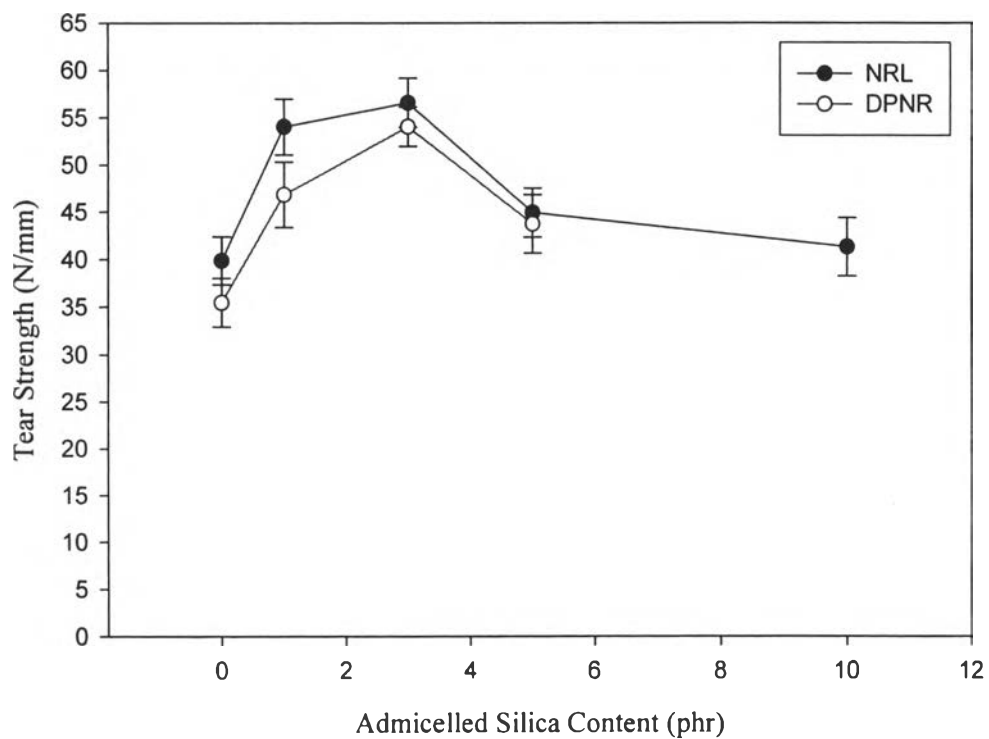
Tensile strength, elongation at break, tear strength and hardness of both vulcanized NRL and DPNR film were determined as shown in Figures 4.5, 4.6, 4.7 and 4.8 respectively. It is noted that the samples made of DPNR with 10 phr admicelled silica were failed to form film on the former such that the tested values were not available.



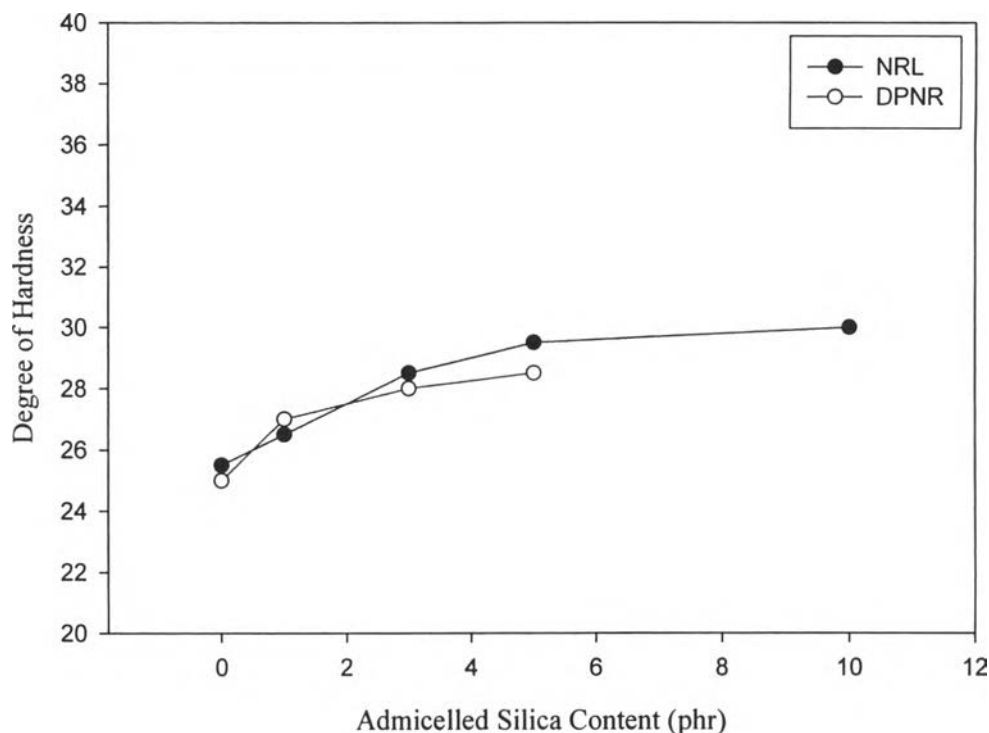
**Figure 4.5** Tensile strength of unaged vulcanized NRL and DPNR films with 10% admicelled silica.



**Figure 4.6** %Elongation of unaged vulcanized NRL and DPNR films with 10% admicelled silica.



**Figure 4.7** Tear strength of unaged vulcanized NRL and DPNR films with 10% admicelled silica.



**Figure 4.8** Hardness of unaged vulcanized NRL and DPNR films with 10% admicelled silica.

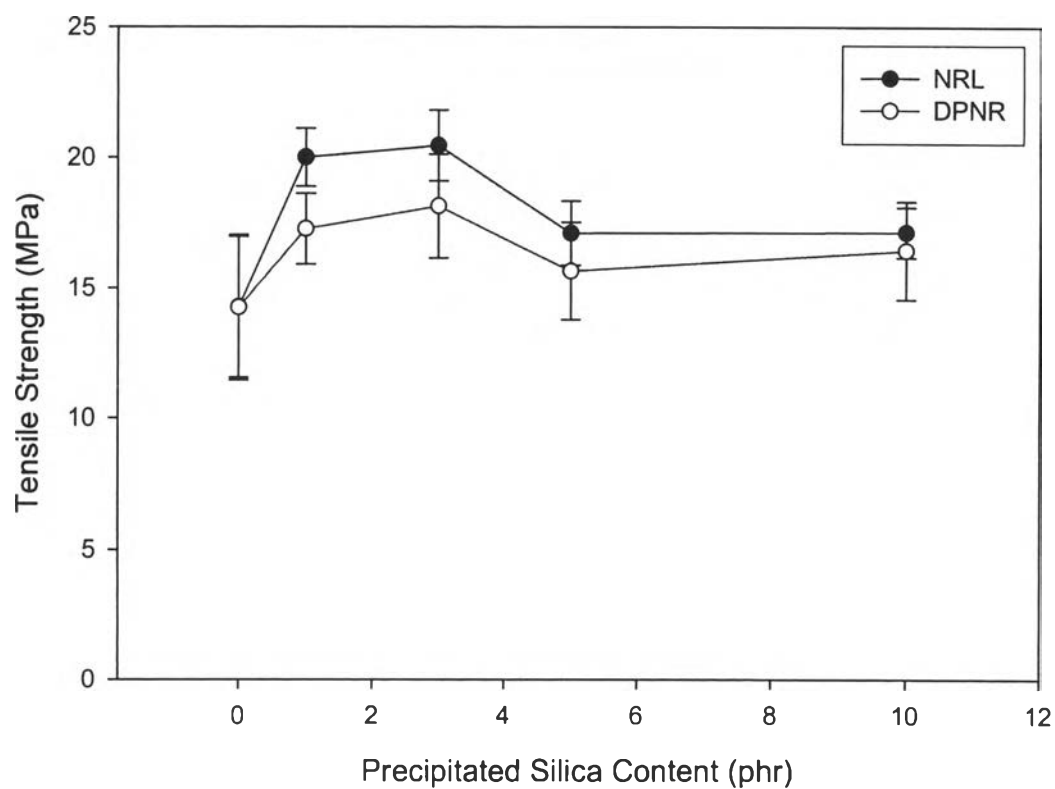
Admicelled silica was different from precipitated silica which can directly link with rubber particle without compatibilizer. Since styrene-isoprene admicellar polymerization made silica particles to have the active site which can directly couple onto rubber surface as same as using silane coupling agent. So, the stiffness will be increased as shown in hardness value. Higher energy was used for breaking rubber chain that may effect on increasing mechanical properties.

The results showed that the values of tensile strength and tear strength increased due to increasing of admicelled silica content in range of 0, 1 and 3 phr which was highest at 3 phr. Then, the values of tensile strength and tear strength were more or less stable when the filler content was more than 3 phr because of a certain amount of accelerator was absorbed by the increasing of silica and was not available as vulcanization accelerator (Plueddemann, 1982).

For the reason about silica absorption, it may occur the damaging of organo-coated silica surface from ball-milling and induce chemical adsorption on the real silica surface.

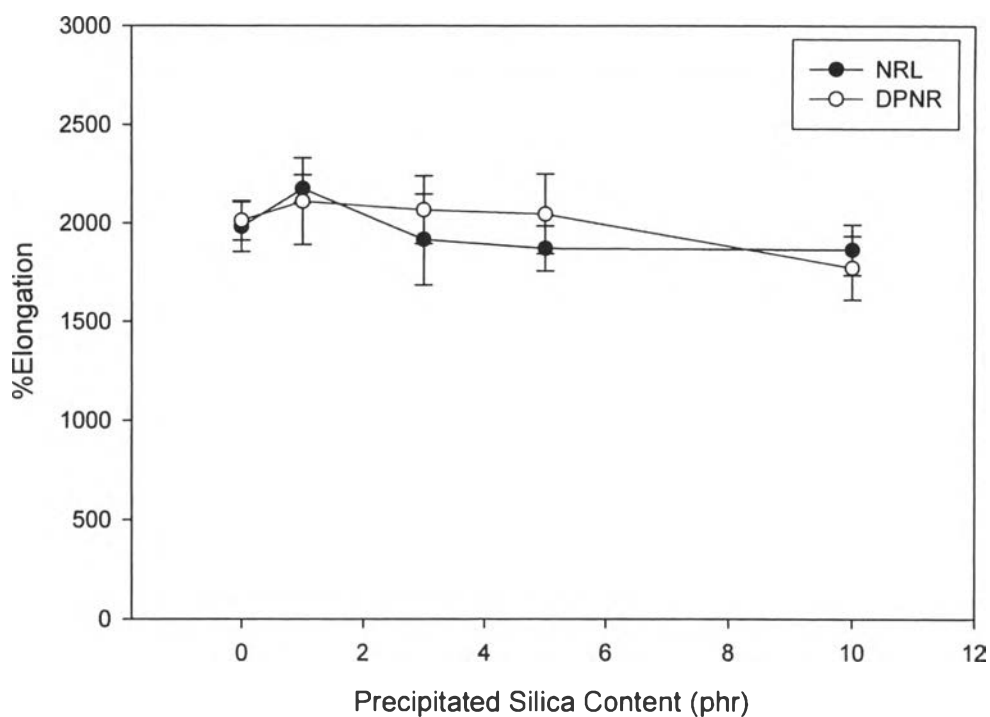
#### 4.1.1.4 Precipitated silica

Tensile strength, elongation at break, tear strength and hardness of both vulcanized NRL and DPNR film were determined as shown in Figures 4.9, 4.10, 4.11 and 4.12 respectively.

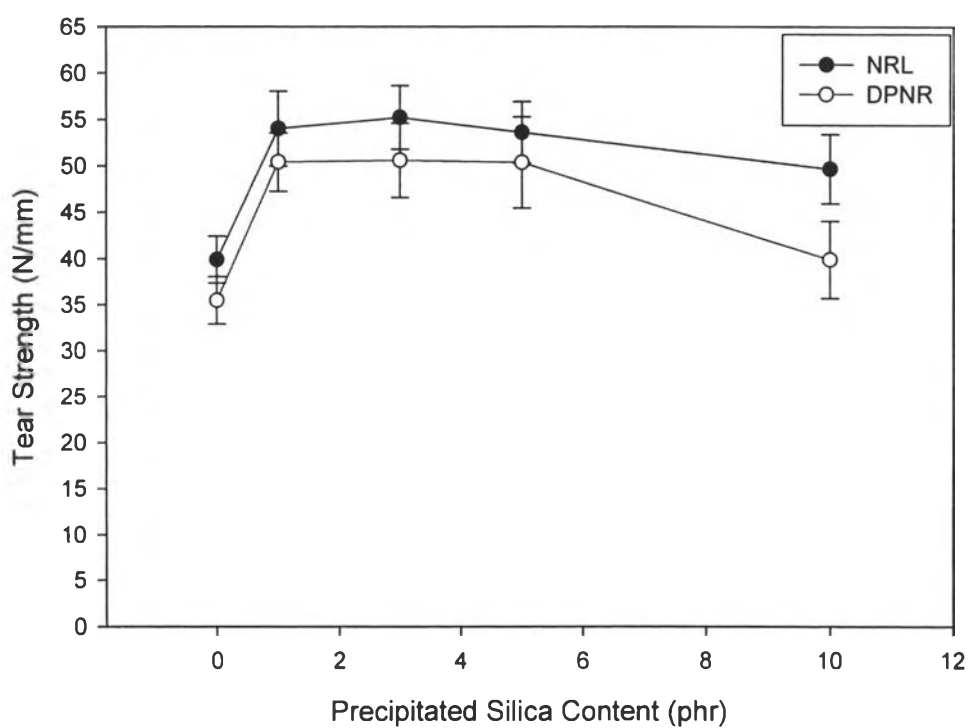


**Figure 4.9** Tensile strength of unaged vulcanized NRL and DPNR films with 10% precipitated silica.

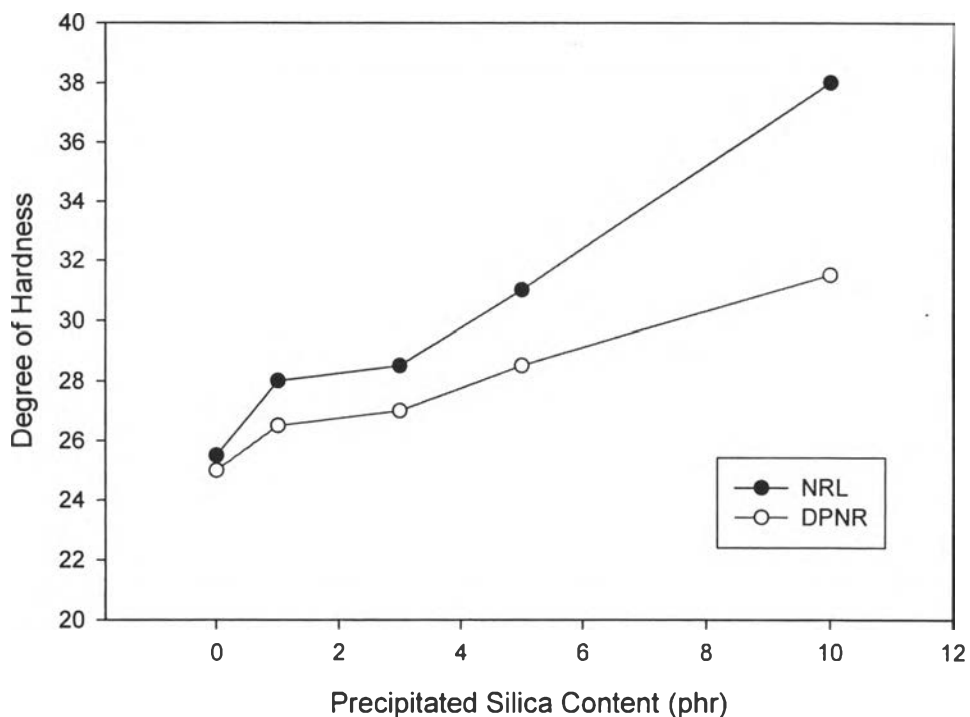




**Figure 4.10** %Elongation of unaged vulcanized NRL and DPNR films with 10% precipitated silica.



**Figure 4.11** Tear strength of unaged vulcanized NRL and DPNR films with 10% precipitated silica.



**Figure 4.12** Hardness of unaged vulcanized NRL and DPNR films with 10% precipitated silica.

In the adding of 10% precipitated silica, SI-69 was used for initiating the chemical reaction via the filler-reactive ethoxisilyl groups with the silanol groups of the silica surface to form the stable siloxane bonds. Also, chemical reactions between the tetrasulfane groups and the elastomer during the vulcanizing process link the silica filler with the rubber matrix to increase rubber stiffness. So, higher energy was used for breaking rubber chain that may effect on increasing mechanical properties which the same trend with adding 10% admicelled silica.

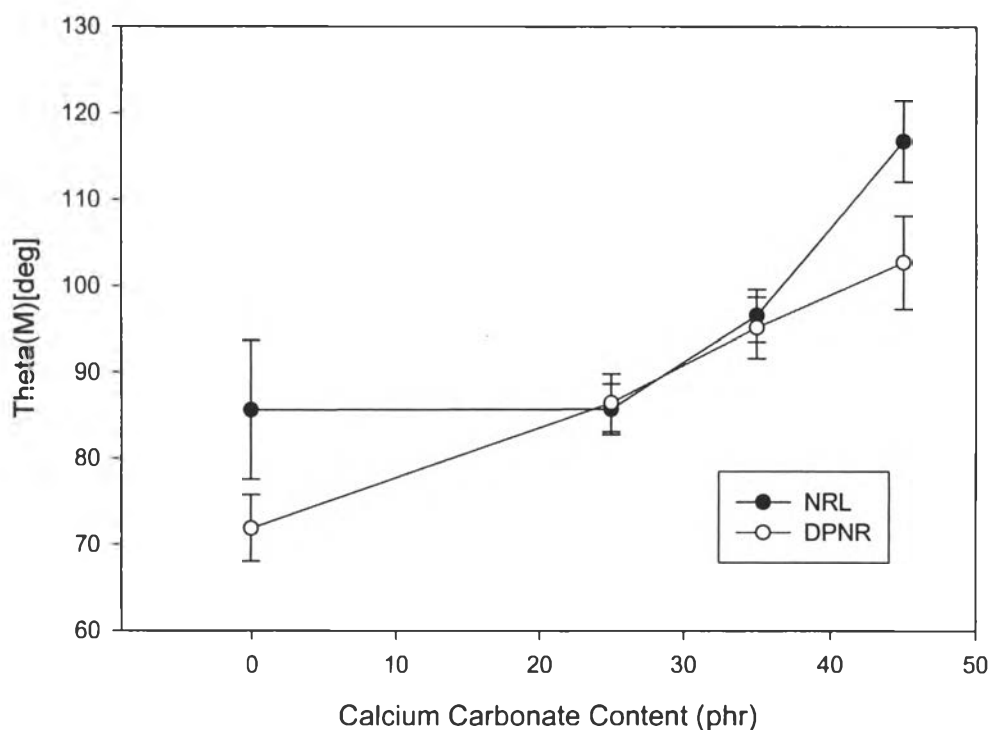
## 4.2 Contact Angle Measurement

Contact angle is observed to study the chemical changes in rubber surface and indicate the surface property. In this work, a slippery surface shows a higher contact angle or a lower friction surface.

### 4.2.1 Calcium Carbonate

Contact angle for water on rubber film showed slippery surface when compared with and without  $\text{CaCO}_3$ . As shown in Figure 4.13, the contact angle increased with increasing the amount of  $\text{CaCO}_3$ .

$\text{CaCO}_3$  was insoluble solid which have high lattice energy. The results indicate that the contact angle increases largely with increasing  $\text{CaCO}_3$  content revealing water is incompatible to the surface of  $\text{CaCO}_3$  filled substrate. A higher contact angle will be obtained. Moreover, It was also observed that  $\text{CaCO}_3$  made the rubber films easy to be detached from the former or increased the desorption of the rubber film.

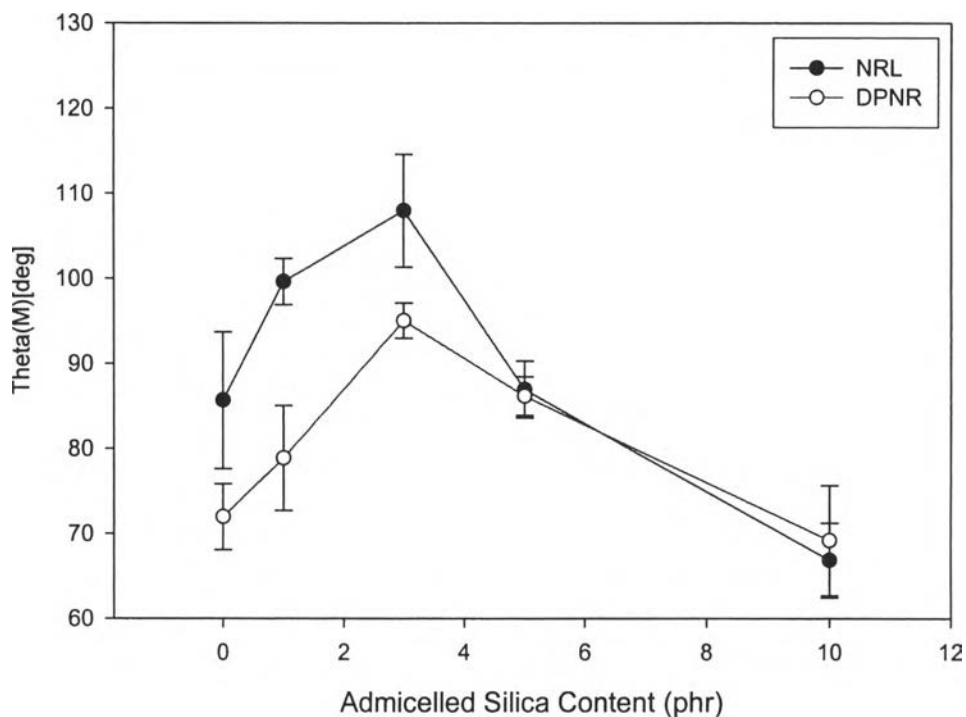


**Figure 4.13** Contact angle of unaged vulcanized NRL and DPNR films with 50% calcium carbonate.

#### 4.2.2 Admicelled Silica

Contact angle for water of both vulcanized NRL and DPNR film were determined by contact angle measurement. As shown in Figure 4.14

Admicelled silica in this work was the coated silica with styrene-isoprene (1:3 molar ratio) by admicellar polymerization. According to the non-polarity of styrene and isoprene particle, it may increase contact angle with increasing the amount of admicelled silica. However, ball-milling can damage the organo-coated silica surface and induce water adsorption on the real silica surface. So, the higher amount of silica, the more water was adsorbed than desorbed.

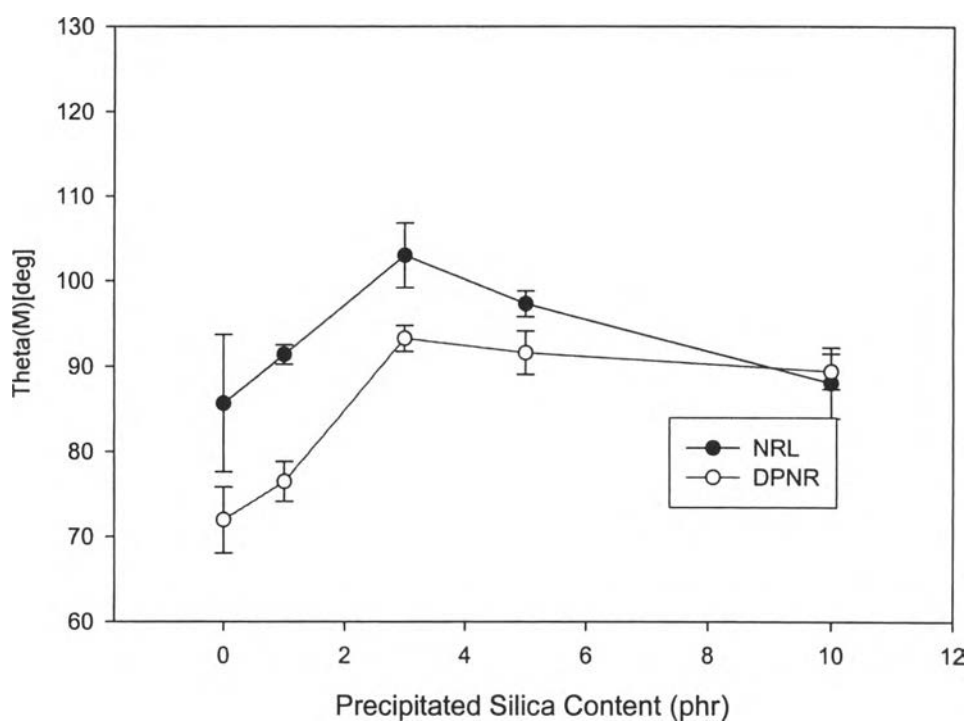


**Figure 4.14** Contact angle of unaged vulcanized NRL and DPNR films with 10% admicelled silica.

### 4.2.3 Precipitated Silica

Contact angle for water of both vulcanized NRL and DPNR film were determined by contact angle measurement as shown in Figure 4.15

The main mechanism of SI-69 to rubber chain and precipitate silica was the linking of rubber chain with silica surface. The structure of SI-69 did not adsorb water thus contact angle was increased with increasing the amount of precipitated silica. However, the failure on the surface of filler in silane-silica preparation which did not treat at high temperature may be only partly activated with SI-69 (Reuvekamp *et al.*, 2001). Also, the higher amount of silica, the more water was adsorbed than desorbed.

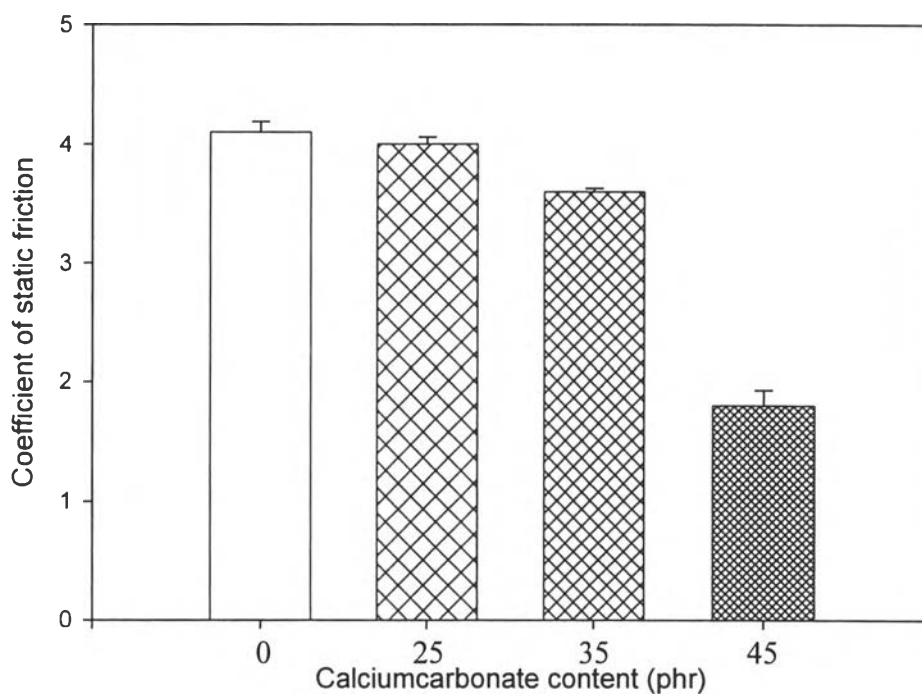


**Figure 4.15** Contact angle of unaged vulcanized NRL and DPNR films with 10%precipitated silica.

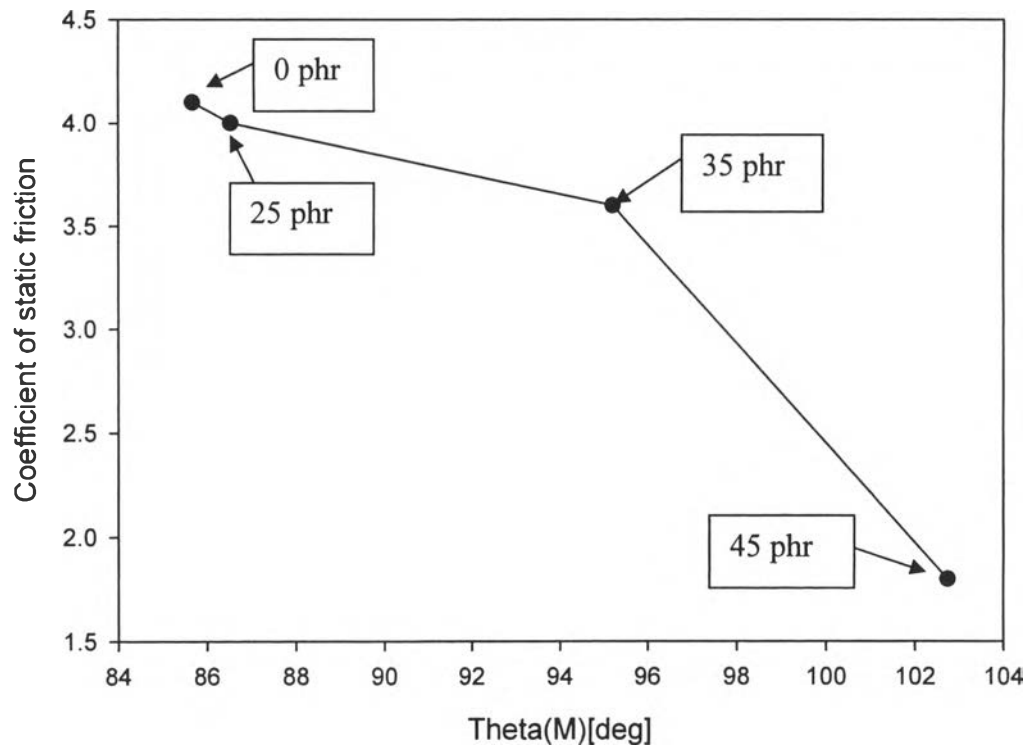
### 4.3 Friction Test

Coefficient of static friction of vulcanized NRL film was determined by friction test as shown in Figure 4.16.

$\text{CaCO}_3$  was only one filler for expecting to reduce surface friction in this work. According to the result of contact angle measurement in  $\text{CaCO}_3$  system, it related with the friction test that higher contact angle corresponds to lower coefficient of static friction with increasing the amount of  $\text{CaCO}_3$  as shown in Figure 4.17. Therefore the surface tackiness of NR could be reduced by the addition of  $\text{CaCO}_3$ .



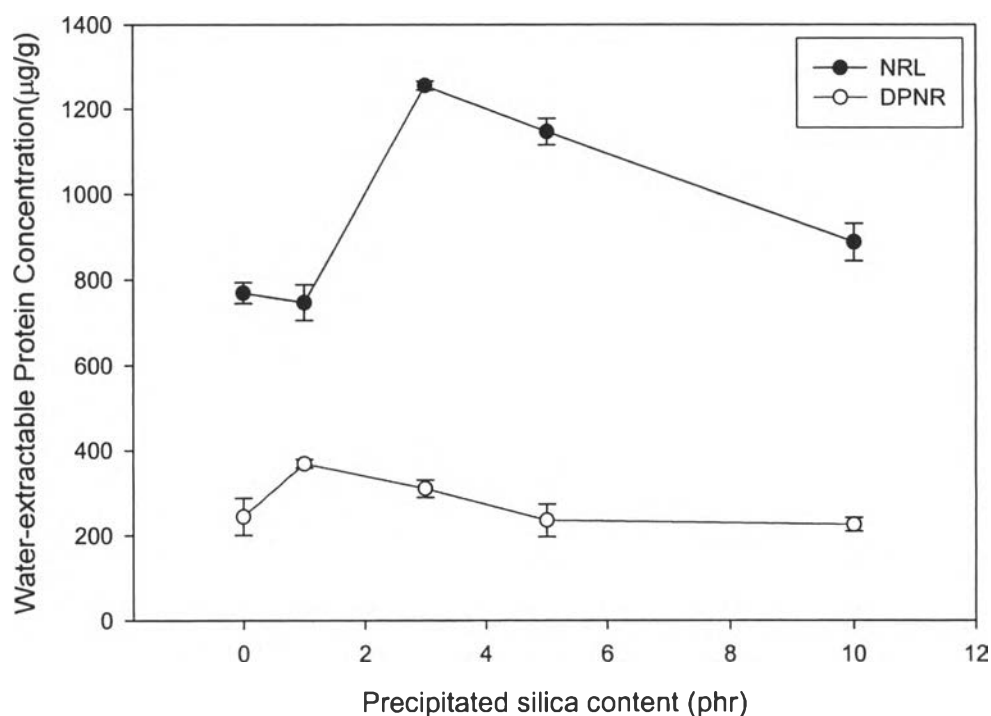
**Figure 4.16** Coefficient of static friction of vulcanized NRL films with 50% calcium carbonate.



**Figure 4.17** The relationship between the contact angle and the friction of rubber surface after compounding the latex with 50%calcium carbonate.

#### 4.4 Water-Extractable Protein Testing

Precipitated silica was only one filler for expecting to remove the water-extractable protein from rubber particle. The result was shown in Figure 4.17.



**Figure 4.17** The total water-extractable protein per gram of vulcanized NRL and DPNR films with 10% precipitated silica.

The result shows that the amount of water-extractable protein quite fluctuates with increasing amount of precipitated silica. These suggest that precipitated silica did not effect on removing water-extractable protein while fumed silica did. These may due to these non-efficient filler cannot substitutes the more polar lipid-protein complex phase which tends to separate from the non-polar hydrocarbon of the rubber phase.

However, the amount of water-extractable protein in DPNR film was obviously less than NRL film.