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EFFECT OF RUBBER BLEND COMPOSITIONS ON THEIR MECHANICAL AND OIL RESISTANCE CHARACTERISTICS

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ABSTRACT:

Polymer blends represent a field of intensive research. In the present work, different rubber blends based on nitrile butadiene rubber, NBR (N3980 – 39% acrylonitrile) have been selected to get a product with high oil resistance and good mechanical properties. The ratio of each component (NBR and chloroprene rubber CR) in NBR – CR blends has been varied and the corresponding vulcanizing systems have been carefully selected.

The rheological properties of the different blends obtained were evaluated. For instance, the scorch time, optimum time of vulcanization have been determined and correlated with the blends composition. The mechanical properties, swelling in toluene and different oils have been evaluated using standard techniques. Besides, the effects of different vulcanizing systems on the mechanical properties and degree of swelling of the vulcanized blends in toluene, as well as the resistance of the blends to the oils has been evaluated.

It has been found that the type of rubber and the composition of blends clearly affect their resistance to oils. In contrast, the type of crosslinking and crosslinking density slightly affect the oil resistance of the corresponding vulcanizates.

KEY WORDS: NBR, CR, rubber blends, vulcanizing system, oil resistance.

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1. INTRODUCTION:

Rubber blends can offer a set of properties that can give them the potential of entering application areas not possible with either of the polymers comprising the blend. So, blending of rubbers is an important route for developing new versatile polymeric materials, the properties of which can be tailored accordingly [1]. The physical properties of polymer blends are generally controlled by many factors, including the nature of the polymer, and blend composition [2].

Accordingly, blending of different types of rubber with each other or with thermoplastics finds increasingly interest and attracts the attention of many investigators [3, 4].

During the last few decades the importance of polymer blends has increased, since it is possible to achieve desirable properties by simple blending of polymers [5 , 6].

A review of rubber blends was published by Ronald [7] and different methods were used for studying polymer-polymer blends [8]. In this sight, the blends of acrylonitrile butadiene rubber and another type of rubber have received wide attention, and the improvement of the performance of the nitrile rubber has been tried by blending with various polymers [9-12].

Nitrile rubbers (NBRs) are oil-resistant elastomers of choice for typical sealing applications [13]. Where, they belong to the class of specialty elastomers that offer a broad range of thermal and oil resistance properties. The polarity imparted by the nitrile group is what creates the hydrocarbon resistance in NBR elastomer [14] to oils, and so, it has been the logical elastomer of selection for service in the oil industry. But, NBR is relatively not resistant to ozone. While, CR is superior in flames retardance, as well as oil and aging resistance [15].

Coran and Patel have developed hot resistance thermoplastic elastomer from blend of NBR with polypropylene (PP) [16]. Blends of NBR with isotactic PP [12,17], chlorosulphonated polyethylene [10], polyacrylic acid [18], polysulphide rubber [19] and polychloroprene [1], have been prepared and investigated.

The object of this paper therefore is to blend NBR with CR for obtaining products having excellent oil resistance in engine and hydraulic oils and to examine the efficacy of these blends.

2. Experimental:

2.1. Materials:

The basic materials used in this work are: Acrylonitrile butadiene rubber (NBR, N3980 – 39% acrylonitrile, Mooney Viscosity (ML-4 (100°C): 80, EINE CHEM, ITALY), Chloroprene rubber (CR, Mooney Viscosity (ML-4 (100°C): 75-90, Bayer, Germany), Zinc Oxide (ZnO, Bayer, Germany), Magnesium Oxide (MgO, Bayer, Germany), sulphur (S, Dammam, KSA), tetramethylthiuram disulphide (TMTD, Bayer, Germany), ethylene thiourea of 2-mercapto imidazole (ETU, Bayer, Germany), 2-

mercaptobenzthiazol (MBT, Bayer, Germany), dibenzothiazyl disulphide (MBTS, Bayer, Germany), N-cyclohexyl-1,2-benzothiazyl sulphenamide (CBS, Bayer, Germany), diortho-tolyl guanidine (DOTG, Bayer, Germany), dicumyl peroxide (DCP, TAlpel, Taiwan).

2.2. Recipes:

Basic formulations used for choose the vulcanizing system for NBR and CR mixtures, and those for determining the best blend compositions of NBR and CR blends are shown in Table 1.

2.3. Techniques and methods:

2.3.1. Mixing and compounding:

Mixing of NBR and CR mixtures were carried out on a laboratory open two roll mill (400 mm. diameters and 600mm. working length). The gear friction of the mill is 1:1.4. The hollow rolls were cooled by using flushing water in order to regulate the temperature not exceeding 60 °C during the different stages of mixing according to ASTM D-15-627. The rubber mixes obtained were sheeted and left for a period of at least 6 hrs before testing.

The addition of ingredients during mixing was carried following the same order and conditions of mixing. The used rubber formulations are given in Table 1.

2.3.2. Rheology measurements:

The cure characteristics of rubber compounds were carried out using an Oscillating Disc Rheometer (ODR) model 4308 from Zwick, Germany. The measured parameters are:

M_L : minimum torque.

M_H : maximum torque.

t_{s2} : Time to (2) units of torque increase above minimum.

M_{c90} : Torque at 90% of full torque development, where:

$$M_{c90} = M_L + [(M_H - M_L) * 90/100] \quad (1)$$

t_{c90} : equivalent to optimum cure time.

Rate of cure:

$$R = [(M_{c90} - M_L) / (t_{c90} - t_L)] \quad (2)$$

Where: t_L is the time of M_L .

These measurements were accomplished according to ASTM D-2084.

2.3.3. Preparation of the test samples:

The rubber mixes were compression molded at 150 ± 2 °C, using an electrically heated hydraulic press at their optimum cure time derived from rheological measurements. The applied hydraulic force during vulcanization attained 50 Kgf/cm^2 .

Dumbbell shaped tensile strength was punched from the compression moulded sheet along the mill grain direction using C-type dumbbell specimens, according to ASTM D-412.

2.3.4. Physico-mechanical properties of the test samples:

* Mechanical properties

The mechanical properties were determined using the tensile testing machine type Zwick 1445 according to ASTM D-412.

*Hardness

The hardness of the samples was measured, according to ASTM D-2240, using a shore 'A' hardness tester of the type HGIB, produced by the VEB Thuringer.

* Resilience

The resilience of the specimens was determined using elasticity instrument type HGIB, produced by the VEB Thuringer, according to ASTM D-1054. It is expressed in percentage %.

All tests were carried out at room temperature (25 ± 2 °C).

2.3.5. Swelling of the test samples:

The equilibrium swelling was carried out according to ASTM D-471. The swelling was performed by immersing the specimens in engine oil Type (50), hydraulic oil Type (37), and toluene at room temperature 25 ± 2 °C, for 24 hours. The variation of apparent percentage of change in mass was calculated. It is expressed as the percentage of the original mass [20-21].

$$\text{Equilibrium swelling \%} = [(W - W_0) / W_0] * 100 \quad (3)$$

Where W_0 : is the initial mass before swelling, W : is the mass after equilibrium swelling.

3. Results and Discussion:

It is necessary to choose the most suitable vulcanization system, namely, accelerators, activators, curing agents combination in order to obtain the desired processing conditions of mixes (scorch time, optimum time of vulcanization, etc) and mechanical properties of rubber vulcanizates. Table 1 shows the four different vulcanizing systems, which were selected to vulcanize NBR rubber, these vulcanizing systems include:

1. Rich sulphur system, as in mixtures (MN1, MN2, and MN3).
2. Low sulphur system, as in mixtures (MN4, and MN6).
3. Non-sulphur system, as in mixtures (MN5, and MN7).
4. Peroxide system, as in mixture (MN8).

Also, Table 1 shows the two different systems, which were selected to vulcanize the CR rubber under investigation, In this case 2 phr and 3 phr of ethylene thiourea ETU were used for MC1 and MC2 mixtures respectively.

Effect of blend compositions of NBR/CR in the ratios has been prepared. The properties of both mixes and vulcanizates have been evaluated. The compositions of the blends are: 100/0, 90/10, 70/30, 50/50, 30/70, 10/90, and 0/100 of NBR/CR blends, as shown in Table 1.

3.1. Rheometric characteristics:

3.1.1. Effect of vulcanization system on rheometric characteristics:

The effects of different vulcanizing systems are given in Table 2. From this table it can be seen that, the scorch and optimum cure time of rubber compounds depend strongly on the used vulcanization system. For instance, the scorch time t_{s2} was very short and attains 1.9 minutes when highly effective vulcanizing system, namely S/TMTD/DOTG system was used. In this case, DOTG as basic accelerator activates TMTD as acidic type and forms "synergism" with high efficiency. This type of vulcanizing system can be used for thin wall articles or latex dipped products.

In contrast the use of CBS in MN1 allows having more safe mixtures, where the scorch time attains 3.4 minutes. This holds true for MBTS also, where scorch time attain 4.6 minutes. Decreasing the sulphur concentration to 0.5 phr and using in addition 2 phr TMTD as in MN4 allows increasing the scorch time. In absence of sulphur as in MN5, the scorch time is very high which reflects the role of sulphur in the vulcanization process. However, the difference between them is only remarkable for TMTD in combination with sulphur.

The rate of vulcanization in this case is also high for these systems (MN3, MN4, and MN6). MN3 formulation possesses the highest rate of vulcanization due to the higher concentration of sulphur and the presence of effective accelerators system (MBTS and TMTD). The higher rate for MN6, however, may be attributed to the possible

synergism occurred between DOTG and TMTD as mentioned before. The synergism alone as in MN7 is not sufficient to give high rate of vulcanization. For mix MN4, the increasing in the concentration of TMTD and the presence of sulphur in the same time is responsible for the high rate.

In case of polychloroprene rubber (CR), the scorch time increases, as expected, with decreasing the concentration of ETU and attains 1.8 min when the concentration of ETU is 2 phr (Mix MC1). The scorch time of the same mix on using 3 phr of (ETU), in the mixture MC2, is shorter and attains 0.4 minutes. The optimum time of vulcanization and also the rate of vulcanization are practically the same for the two concentrations of ETU. Accordingly, for further study we are going to use ETU in concentration 2 phr.

3.1.2. Effect of the compositions of NBR/CR blends on rheometric characteristics:

In case of NBR/CR blends, we selected the proper vulcanizing system calculated according the weight fraction of each type in the blends. The results obtained for the different rubber blends are given in Fig.1 and in Table 3. It was found that, the scorch time value is dependent on the CR content of the different blends. Whereas, the increase in CR concentrations decreases the scorch time slightly for (10/90) NBR/CR. The optimum cure time value is also dependent on the CR content of the different blends. Thus, an increase of CR content from (0%) to (100%) will result in a decrease in scorch time and an increase of optimum cure time values.

3.2. Physico-mechanical properties:

3.2.1. Effect of vulcanization system on physico-mechanical properties:

It is known that the mechanical properties of rubber depend on the type and concentration of crosslinks. Accordingly, the results obtained showed that, the physico-mechanical properties of rubber vulcanizates depend markedly on the system of vulcanization used. Table 2 presents the tensile strength, modulus (100%), and modulus (200%) for all mixtures.

From Table 2 it can be seen that for NBR mixtures, modulus at 100% of mixtures MN3 and MN8 were (34 Kgf/cm² and 31 Kgf/cm²) respectively. These mixtures had the highest modulus 100% values in comparison with others. On the other hand, the mixtures MN3 and MN8 had the highest modulus 200% values respectively, in comparison with others.

For CR mixtures, modulus at 100% and modulus 200% of mixture MC1 is practically the same as that of MC2.

Table 2 shows that the mixture MN3 had the lowest elongation at break 379%, in comparison with other NBR mixtures. Whereas, the mixture MC1 had the lowest elongation at break value 425%, for CR mixtures.

From Table 2, the mixture MN3 had a low value of the permanent set 3%, and a high value of resilience 22%, in comparison with other NBR mixtures. Whereas, the mixture MC1 had the lowest permanent set value 1%, but had a low resilience value 35%, for CR mixtures.

Summarizing, it can be concluded that the type of vulcanizing system slightly affect the hardness and the resilience of the corresponding vulcanizates. The permanent set values are almost the same except for the mixes not containing sulphur (MN5 and MN7) which indicates that these samples are under cured. This is confirmed by the high elongation at break and lower values of modulus.

3.2.2. Effect of NBR+CR blends compositions on physico-mechanical properties:

It was found that, the physico-mechanical properties of rubber blends depend markedly on the CR content. Fig.2 presents the tensile strength, modulus (100%), and modulus (200%) properties of all NBR/CR blends. When the CR content increases the tensile strength property increases governed by the rule of additives as the tensile strength of the blend equals the weight fraction of each components multiplied by the value of each property. This is valid only when the two components are compatible with each other.

The results obtained showed the relationship between the weight fraction of CR in the blend and the tensile strength is linear and the equation represents this relationship between CR content in the blend and the tensile strength value is give by the following equation:

$$\text{Tensile Strength value of the blend} = 79.4 x + 131.9, R^2 = 0.93 \quad (4)$$

Where: x is the weight fraction of CR in the blend, and R^2 is the square of the correlation coefficient reflecting the linearity.

Also, increasing CR leads to an increase in the modulus (100%) and modulus (200%) till an optimum value at (50/50) NBR/CR blend, then start to slightly decrease.

In this case the relation is not linear and can be given by the following equation:

$$\text{Modulus of blend at 200\% value} = 99.9 x - 98.6 x^2 + 70.6, R^2 = 0.94 \quad (5)$$

Where: x is the weight fraction of CR content in the blend, and R^2 is the square of the correlation coefficient.

The equation which gives the modulus at 100% is:

$$\text{Modulus of blend at 100\% value} = 56.2 x - 59.1 x^2 + 34.4, R^2 = 0.95 \quad (6)$$

Where: x is the weight fraction of CR content in the blend, and R^2 is the square of the correlation coefficient.

These two equations indicate non-linearity which means that NBR is not compatible with CR which is more accurate than in case of tensile strength as the last property depends on many other factors and can not be taken as real indication of compatibility.

Fig.3 shows that, the elongation at break of rubber blends depends slightly on the CR content. When the CR content increases the elongation at break slightly decreases for (30/70) NBR/CR blend, then the elongation increases.

From Fig.4, it is shown that the resilience of rubber blends depends markedly on the CR content. When the CR content increases the resilience decreases for (10/90) NBR/CR blend, then the elongation markedly increases. For CR rubber only, it has resilience value 35.

In contrast, no significant change in the permanent set with increasing of the CR content has been observed. When the CR content increases the permanent set slightly decreases.

The hardness was found to be slightly affected by CR content in the NBR/CR blends, whereas the increase in CR content slightly increases the hardness for (50/50) NBR/CR blend then decreases, as shown in Fig.5. For (50/50) NBR/CR blend, it had relatively high hardness values 78.

3.3. Swelling properties:

3.3.1. Effect of vulcanization system on swelling properties:

It is obvious from Table 2 that, the mixtures MN2, MN3, MN5, MN7, MN8 had excellent engine oil resistance, whereas, the mixtures MN3- MN7 had excellent hydraulic oil resistance for NBR formulations. The degrees of the swelling of the mixture MC1 in both (engine and hydraulic oils) and in toluene is lower than those of the MC2.

3.3.2. Effect of NBR+CR blends compositions on swelling properties:

It is obvious from Fig.6, that, the degree of equilibrium swelling in engine oil is slightly affected by CR content, whereas the increase in CR content slightly increases the swelling. The equation which gives the swelling in engine oil with 0.92 value of the square of the correlation coefficient, was:

$$\text{Swelling in Engine oil \%} = 0.48 x^2 - 0.30 x + 0.02, \quad R^2 = 0.92 \quad (7)$$

Where: x is the weight fraction of CR content in the blend, and R^2 is the square of the correlation coefficient.

In contrast, the degree of swelling in hydraulic oil is strongly affected by CR content, where the increase in CR content strongly increases the degree of swelling. The equation which gives the swelling in hydraulic oil with 0.9 value of the square of the correlation coefficient, was:

$$\text{Swelling in Hydraulic oil \%} = 1.83 x^2 - 0.62 x + 0.04, R^2 = 0.9 \quad (8)$$

Where: x is the weight friction of CR content in the blend, and R^2 is the square of the correlation coefficient.

The degree of swelling in toluene is clearly affected by CR content, whereas the increase in CR content increases the equilibrium swelling increase, as shown in Fig.7. The related equation was:

$$\text{Swelling in Toluene \%} = 54.3 x + 52.9, R^2 = 0.97 \quad (9)$$

Where: x is the weight fraction of CR content in the blend, and R^2 is the square of the correlation coefficient.

This equation (9) is a linear one with 54.3 value of the slop, and 0.97 the value of the square of the correlation coefficient. The positive value of the slop indicates that the equation represents an incremental manner of swelling property with increasing the CR concentration.

4. CONCLUSION:

From the above mentioned results the following conclusions can be derived:

1. The optimum properties of NBR formulations were found for the mixture MN3, where the vulcanizing system used was: S 2.5 phr + TMTD 0.7 phr + MBTS 0.5 phr, was the more efficient used system than the others. Also, for obtaining optimum properties of CR mixtures, the vulcanizing system should be: ETU 2 phr for MC1 formulation.
2. Increasing the CR content leads to an increase in the following properties: cure time, tensile strength, elongation at break, resilience, and the degree of swelling in hydraulic oil and toluene; while, the modulus (100%) and modulus (200%) were increase for (50/50) NBR/CR blend then decrease. In this case, the rest of properties are slightly affected. Thus, the optimum concentrations of NBR and CR in the blend will determined according to the required properties.

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FIGURES AND TABLES

Table 1: Formulations of NBR and CR with different vulcanizing systems and different blend ratios

BLEND CODE	MN1	MN2	MN3 (B1)	MN4	MN5	MN6	MN7	MN8	MC1 (B7)	MC2	B2	B3	B4	B5	B6
NBR	100	100	100	100	100	100	100	100	-	-	90	70	50	30	10
CR	-	-	-	-	-	-	-	-	100	100	10	30	50	70	90
st.acid	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
ZnO	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
ZA 4010	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
MgO	-	-	-	-	-	-	-	-	4	4	0.4	1.2	2	2.8	3.6
C.B.(GPF)	45	45	45	45	45	45	45	45	45	45	45	45	45	45	45
DBP	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
CBS	0.7	-	-	-	-	-	-	-	-	-	-	-	-	-	-
MBT	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-
MBTS	-	1	0.5	-	1	-	-	-	-	-	0.45	0.35	0.25	0.15	0.05
DOTG	-	-	-	-	-	1	1	-	-	-	-	-	-	-	-
TMTD	-	-	0.7	2	2.5	3	3	-	-	-	0.63	0.49	0.35	0.21	0.07
S	2.5	2.5	2.5	0.5	-	0.5	-	-	-	-	2.25	1.75	1.25	0.75	0.25
ETU	-	-	-	-	-	-	-	-	2	3	0.2	0.6	1	1.4	1.8
DCP	-	-	-	-	-	-	-	3	-	-	-	-	-	-	-

Table 2. The properties of different NBR and CR mixes and vulcanizates.

Mixtures Code		MN1	MN2	MN3	MN4	MN5	MN6	MN7	MN8	MC1	MC2		
Rheometric Properties	Ts2	[min]	3.4	4.6	3.7	4.3	11.6	1.9	2.9	3.3	1.8	0.4	
	Tc90		21	15	7	9	17.5	9.5	22	21.5	15	14	
	R		0.197	0.18	0.876	0.573	0.155	0.42	0.074	0.066	0.233	0.23	
Physico-mechanical Properties	Tensile Strength	[Kg/cm ²]	163	187	136	148	142	122	172	178	223	238	
	Modulus 100%		31	28	34	21	13	24	16	31	39	34	
	Modulus 200%		61	52	68	37	18	45	25	66	87	74	
	Elongation		%	459	603	379	701	1478	505	1213	411	425	496
	Permanent Set		%	3	4	3	5	25	0	21	2	1	4
	Hardness		'A'	68	68	71	62	55	64	57	67	69	68
	Resilience		%	19	18	22	22	20	22	21	20	35	43
Swelling in:	Engine oil (50)	%	0.8	0	0	0.4	0	0.3	0	0	0.2	0.6	
	Hydraulic oil (37)		0.2	0.6	0	0	0	0	0	0.4	1.5	2.4	
	Toluene		64.6	63.3	55.5	57.1	75.8	59.7	73.3	51.1	108.9	113	

Table 3. The overall properties of different NBR and CR blends.

Blends Code (NBR/CR)		B1 (MN3)	B2	B3	B4	B5	B6	B7 (MC1)
Rheometric Properties	Ts2	3.7	1.6	1.3	1.2	1.2	1.2	1.8
	Tc90	7	12	9.4	15	16.3	17.6	15.5
	R	0.876	0.54	0.645	0.376	0.324	0.257	0.225
Physico-mechanical Properties	Tensile Strength	136	142	147	176	185	193	223
	Modulus 100%	34	40	46	49	43	38	39
	Modulus 200%	68	84	90	96	92	81	87
	Elongation	379	324	304	355	364	411	425
	Permanent Set	3	2	3	2	2	2	1
	Hardness	71	73	74	78	73	72	69
	Resilience	22	16	17	25	25	32	35
Swelling in:	Engine oil (50)	0	0	0	0	0	0.13	0.22
	Hydraulic oil (37)	0	0.02	0.08	0.26	0.52	0.66	1.52
	Toluene	55.5	58.4	70.4	74.5	87.4	105.4	108.9

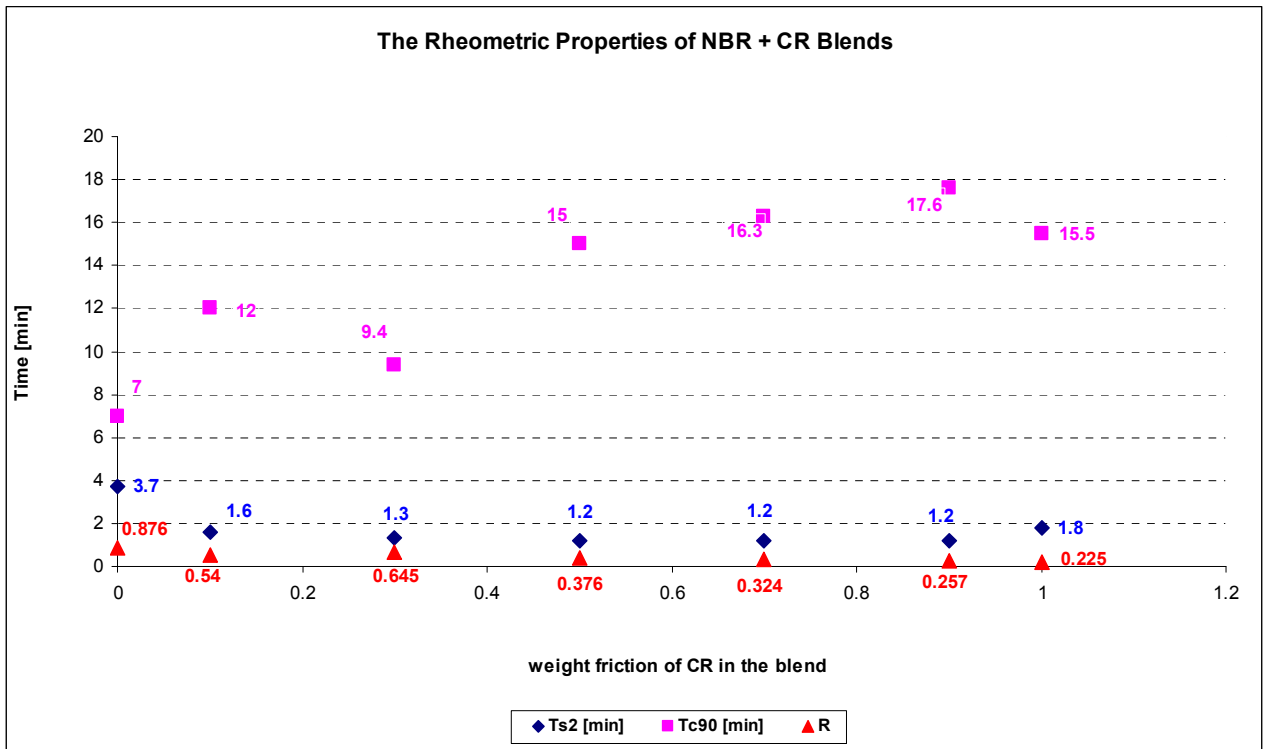


Fig.1. Rheometric characteristics of the different NBR/CR blends.

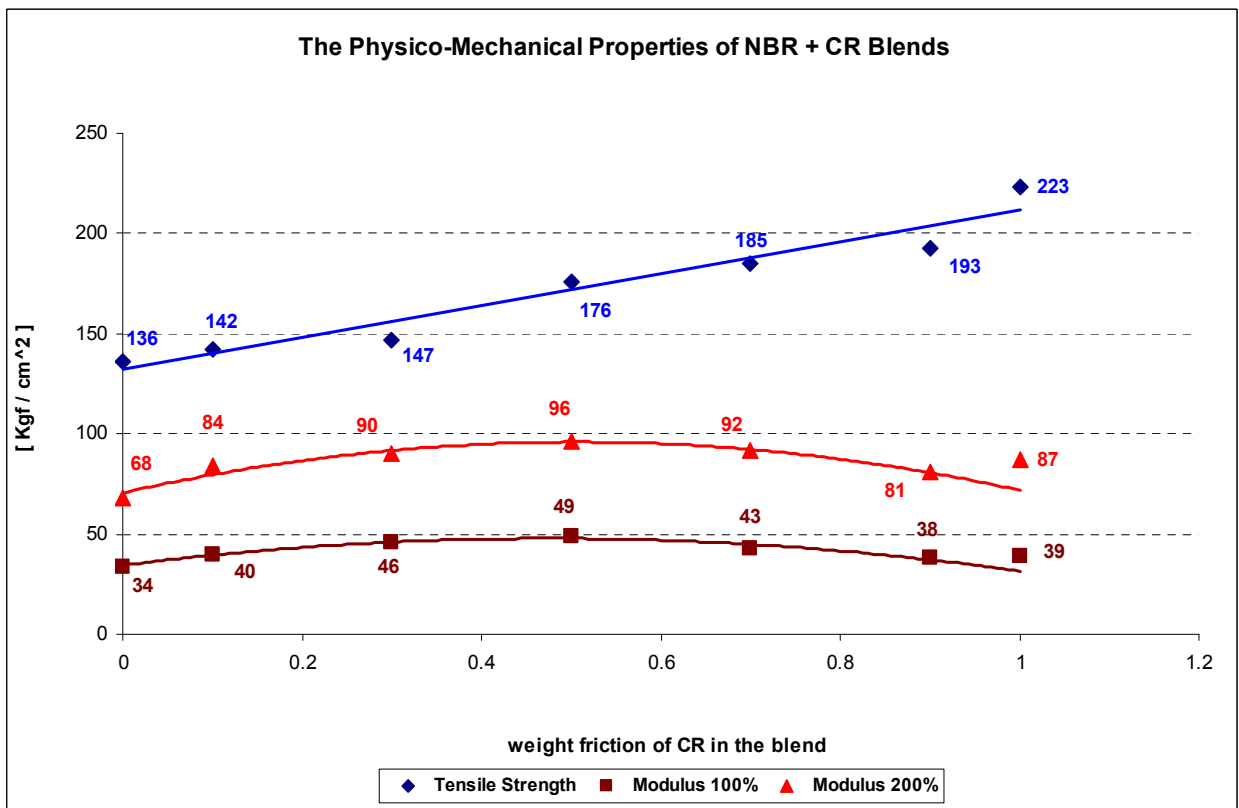


Fig.2. Stress-Strain properties of the different NBR/CR blends.

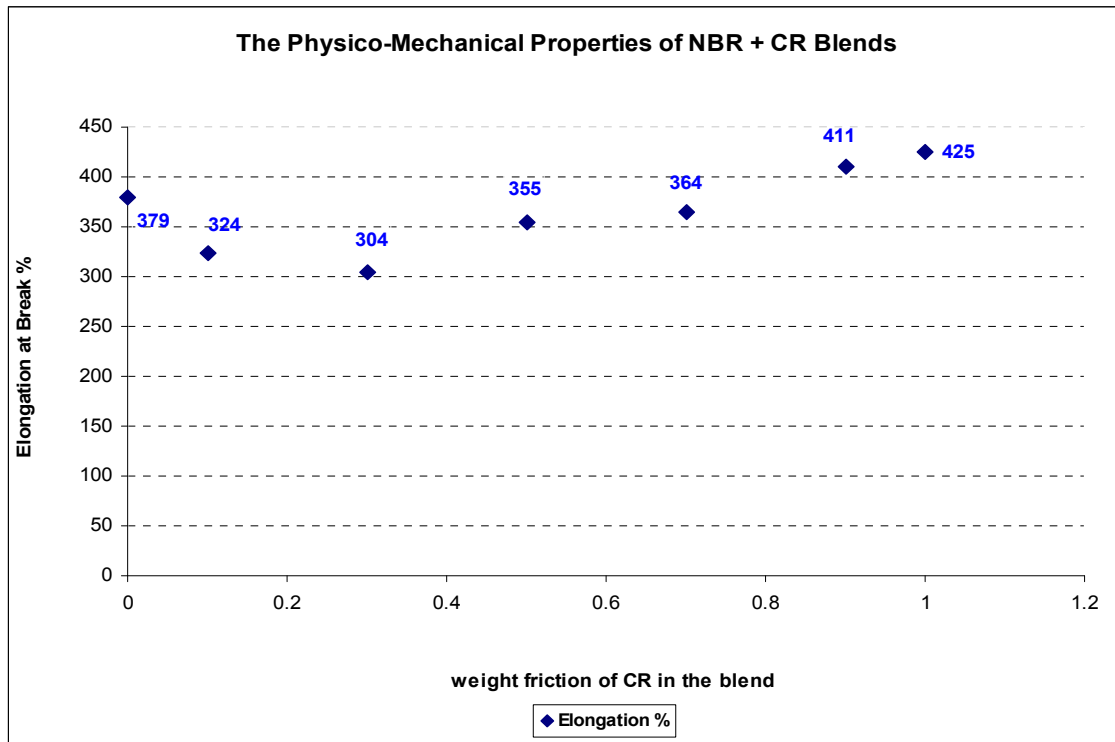


Fig.3. Elongation at break property of the different NBR/CR blends.

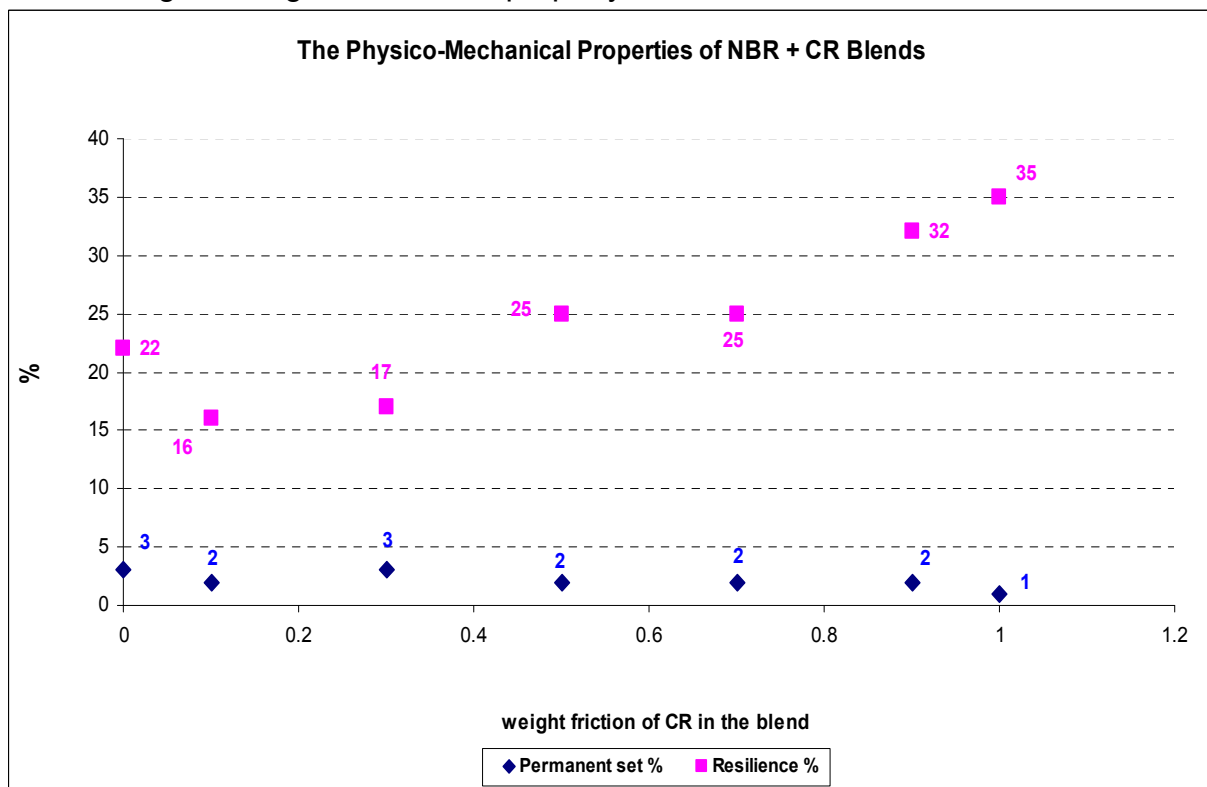


Fig.4. Resilience and permanent set properties of the different NBR/CR blends.

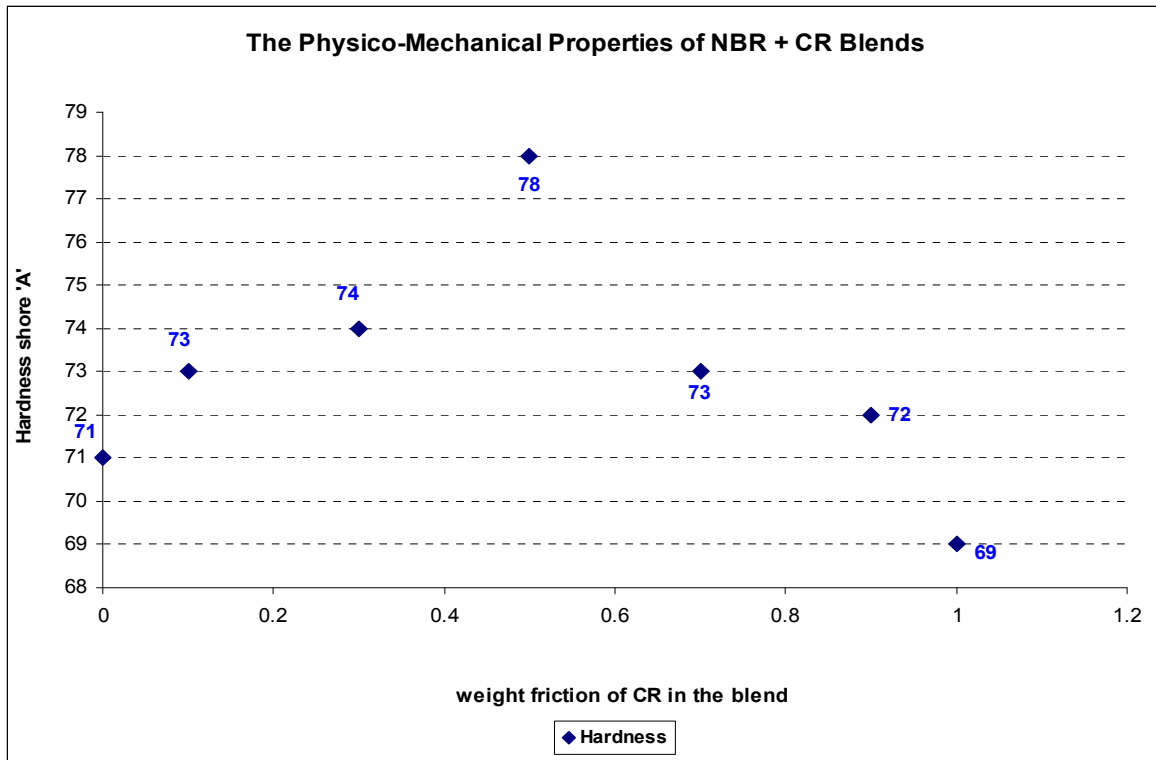


Fig.5. Hardness property of the different NBR/CR blends.

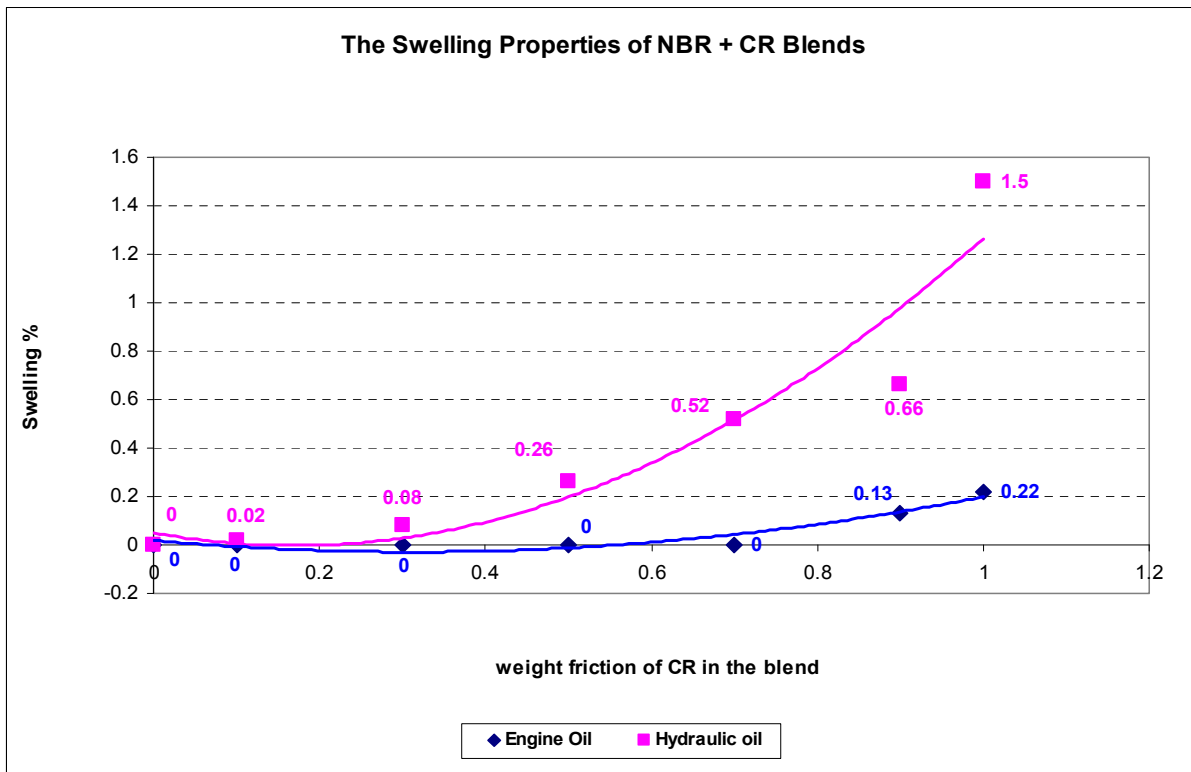


Fig.6. Swelling property in engine and hydraulic oils of the different NBR/CR blends.

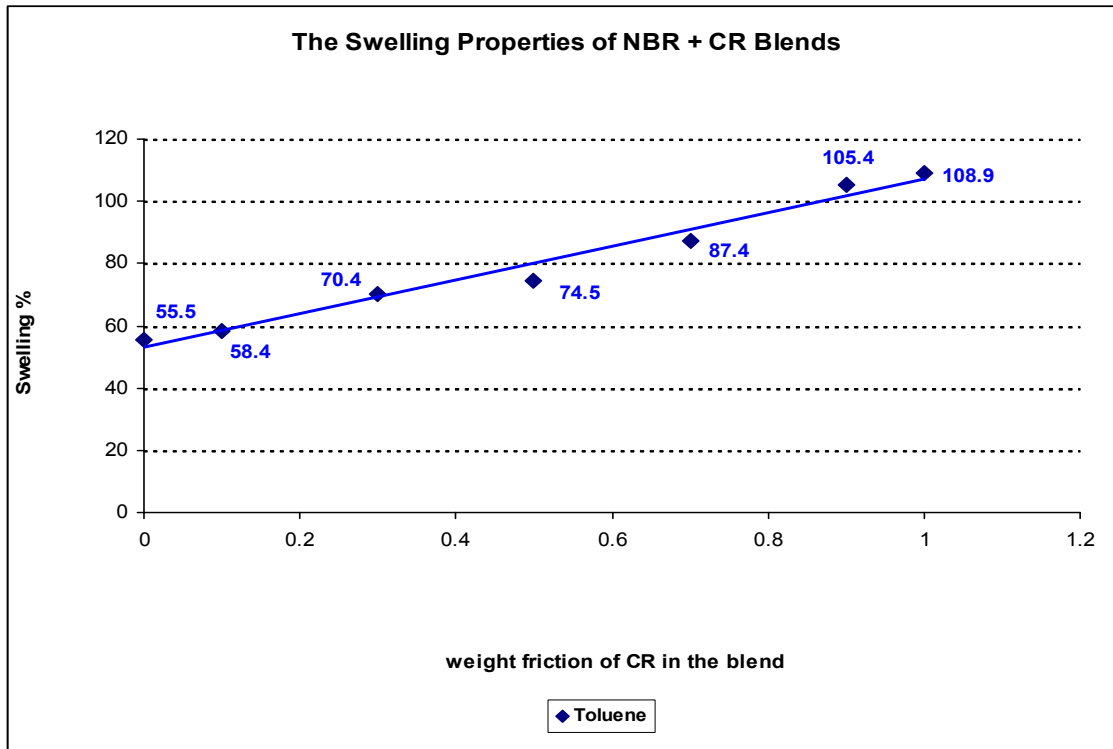


Fig.7. Swelling property in toluene of the different NBR/CR blends.