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Ageing and Oil Resistance of Fiber-Reinforced CR/NBR Blends

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Ageing and Oil Resistance of Fiber-Reinforced CR/NBR Blends

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Nitrile rubber (NBR) as an excellent oil resistant was blended with the thermal resistant polychloroperene rubber (CR) to obtain rubber product with satisfactory thermal and oil resistance. The effect of curing system and blend ratios upon the physico-mechanical, swelling and ageing properties were studied for polyester reinforced vulcanizates. The obtained results were compared with the grafted and ungarfted viscose fibers.

Keywords: CR/NBR blends; grafting; vinyl monomers; viscose fiber; PET fiber; thermal stability; swelling resistance

INTRODUCTION

Fiber reinforced rubber composites impart useful mechanical properties such as stiffness, strength, modulus, and damping in addition to processing advantage. [1-2] Therefore, the major application of fiber reinforcement is directed toward the production of hose and complex shaped mechanical parts. [2] The reinforcement effect of short fibers is governed by a number of factors; fiber type, length and loading, adhesion between fiber and matrix, oreintation and breakage during mixing etc.. The proper choice of bonding agents has also been the subject of many investigators. [4-2] The properties and performance of short fiber-reinforced rubber composites have been investigated. [10, 11] It has been found that the addition of fiber markedly reduces maximum swelling and emails an increase in material stiffness. [12]

In the present research, CR/NBR blend and its polyester (PET) fiber reinforcement were examined in terms of physico-mechanical properties and swelling behaviour in motor oil and brake fluid. The effects of different curing systems and various blend ratios were also investigated after and before thermal ageing.

MATERIALS

- Krynac- N- 3345 (Nitrile rubber "NBR") of 33% acrylonitrile content and 45 Mooney viscosity.
- Neoprene WRT (Polychloroprene rubber "CR"). Product of Bayer company.
- Polyester fibers (PET) of 38 mm length and 1.5 denier. Product of Misr company for artificial silk - Kafer El-Dawar, Egypt.
- Motor oil: Esso Extra multi grade oil 20W-50 AP/SF/CC (Exxon).
- Hydraulic brake fluid: super 105. Product of Lockheed, England.
- · Compounding ingredients are of commercial grades used in industry.

TECHNIQUES

Mixing^[13]: Mixing was accomplished on a laboratory two roll mill; 170 mm diameter and 300mm working distance. The speed of slow roller was 24 rpm with 1:1.25 gear ratio. Care was taken to assure that the fibers orientation is in the grain direction.

Rheometric study: The cure characteristics of the rubber mixes were obtained at 152±1°C using a Monsanto Oscillating Disc Rheometer model-100, (ASTM). [14]

Vulcanization: The uncured rubber sheets (2 mm thickness) were vulcanized in a hydraulic press vulcanizator at 152 ± 1 °C under 4 MPa pressure.

Physico-mechanical Properties: Tensile strength, elongation at break, 100% modulus and Young's modulus were determined using tensile testing machine, Zwick 1425, Germany, according to ASTM method.^[15]

Swelling of rubber vulcanizates in liquids ^[16]: Swelling tests in toluene was carried out at room temperature (25°C) for 48 hours. Swelling tests in motor oil and brake fluid were conducted at 100 ± 1 °C for 1, 2, 4, 6 and 7 days, in a good airiated oven.

Thermal ageing Procedure: Accelerated thermal ageing was carried out in an oven supported with air circulation at 90 ± 1 °C for periods of 2, 4, 6 and 7 days, according to ASTM method. [17]

RESULTS AND DISCUSSION

Blending of CR and NBR and their PET fiber composite

In the present investigation; CR, NBR and thier 50/50 blend were prepared on a two roll mill. Also, polyester (PET) fiber-reinforcement of CR/NBR (50/50) blend was investigated. It is noted that the adhesion system, which consists of hydrated silica, resorcinol and hexamethylene tetramine (HRH) was added to the blend mix. PET fibers (20 phr) were incorporated as one of the rubber ingredients during the mixing process. The orientation of fibers was achieved along the direction of the cylinder rotation during the roll mill operation.

Table 1 shows that CR vulcanizate has higher maximum torque (M_H) and longer cure time (tc90) than NBR vulcanizate. However, CR/NBR blend vulcanizate has a medium value of maximum torque, but short cure time as that of NBR vulcanizate. The maximum torque increases, by incorporating the adhesion system and PET fibers, successively, into CR/NBR blend mix. While, the cure time and the scorch time (ts2) decrease due to the accelerating effect of the adhesion system on the vulcanization of CR/NBR blend.

It is obvious from Table 1 that CR/NBR blend vulcanizate has a tensile strength value which is approximately equal to those values of the individual CR and NBR vulcanizates. While that blend has medium values of elongation at break, 100%modulus, Young's modulus and swelling in toluene, when compared to the individual rubber vulcanizates.

Tensile strength, elongation at break and swelling in toluene values of CR/NBR blend decrease by the addition of HRH adhesion system, then further decrease takes place by incorporation of PET fibers. On the other hand, 100% modulus and Young's modulus values increase by addition of HRH adhesion system, followed by further increase; due to the incorporation of PET fibers. This may be attributed to the in situ formation of resorcinol formaldehyde resin during vulcanization, and due to the rigidity PET fibers. Also, it is clear that tensile strength, 100% modulus and Young's modulus values of PET fiber composite measured in the longitudinal (L) direction are greater than those measured in the transverse (T) direction. But L elongation at break is lower than T elongation. This indicates that the fibers were well oriented in the grain direction of rubber mix.

Table 1 Formulations, rheometeric characteristics and physico-mechanical properties of CR, NBR, their blend and composite

Rubber ingredients	Mi	M2	M3	M4	MS
CR	100	0	50	50	50
NBR	0	100	50	50	50
Stearic acid	2	2	2	2	2
ZuO	5	5	5	5	5
IPPD	1	1	1	i	1
HAF	40	40	40	40	40
Proc. oil	5	5	5	5	5
Histl			_	5	5
Resorcinoi		_	_	5	5
HMT		_	-	3.2	3.2
PET*	-				20
MgO	4	-	2	2	2
ETU	0.5	_	0.25	0.25	0.25
MBT	_	1.5	0.75	9.75	0.75
S		1.5	0.75	0.75	0.75
Rheometric characteristics	at 152± 1°C				
Minimum torque (ML), dN.m.	7	5	9	10	13
Maximum torque (Ma), dN.m.	71	37	59	80	89
Cure time (tem), min.	40.5	25.0	25.5	22.5	22.0
Scorch time (ts2), min.	2.00	4.25	2.50	1.50	1.25
Cure rate index (CRI), min. 1	2.6	4.8	4.3	4.7	4.8
Mechanical properties at 25	5 °C				
Tensile strength, MPa (L)	16.2	15.9	16.4	15.5	10.5
Teusile strength, MPa (T)	16	15.5	16	15.3	8.8
Elongation at break, % (L)	276	660	415	310	215
Elongation at break, % (T)	270	675	420	315	240
100% modulus, MPa (L)	2.8	0.5	1.3	3.2	7.6
100% modulus, MPa (T)	2.7	0.5	1.2	3.0	5.1
Young's modulus, MPa (L)	6	2.4	3.97	4.9	4.9
Young's modulus, MPa (T)	5.5	2.1	3.5	4.2	4.0
Swelling in toluene, for 48 h	rs				
Weight swell, %	149	207	177	138	123

Polyethylene terphthalate (polyester) short fibers.
(L) = Longitudinal direction
(T) = Transverse direction

The physico- mechanical properties of the prepared vulcanizates were determined after thermal ageing for different time periods. Figures 1-3 show that blending of CR&NBR resulted in a vulcanizate with medium physico-mechanical properties when compared to those values obtained for the individual CR and NBR

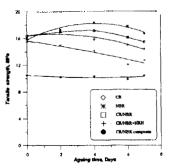


Figure 1 Tensile strength vs. ageing time, at 90 C, of CR, NBR and 50/50 CR/NBR blend and its PET reinforced vulcanizate.

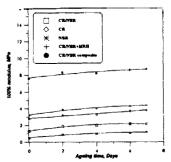


Figure 3 100% modulus vs. ageing time at, 90 C, of CR, NBR and 50/50 CR/NBR blend and its PET reinforced valcanizate.

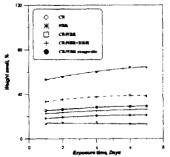


Figure 5 Swelling in brake fluid vs. exposure time, at 100 C, of CR, NBR and 50/50 CR/NBR blend and its PET reinforced vulcanizate.

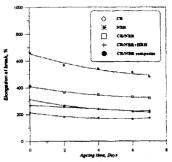


Figure 2 Elongation at break vs. ageing time, at 90 C, of CR, NBR and 50/50 CR/NBR blend and its PET reinforced vulcanizate.

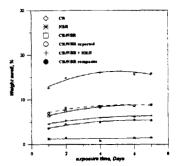


Figure 4 Swelling in motor oil vs. exposure time, at 100 C, of CR, NBR and 50/50 CR/NBR blend and its PET reinforced vulcanizate.

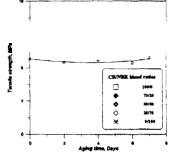


Figure 6 Tensile strength in L direction vs ageing time at 90 C, of the composites (20 phr PET) with various CR/NBR blend ratios.

vulcanizates, after thermal ageing. Also, a balance of properties is obserbved upon blending. The blend vulcanizate reinforced with PET fibers shows no change in tensile strength, a slight decrease in elongation at break and a slight increase in 100% modulus, during the ageing periods. Thus, the reinforced CR/NBR vulcanizate has good thermal stability.

Figuers 4 and 5 show that swelling of NBR vulcanizate is the least in motor oil, while swelling of CR vulcanizate is the least in the hydraulic brake fluid. On the other hand, CR/NBR blend vulcanizate shows medium swelling values, in both motor oil and brake fluid, for all swelling periods. These swelling values are less than the expected values obtained by applying the simple additive rule to CR and NBR swelling values. In other words, swelling values in brake fluid of the blend show negative deviation from the additive rule indicative of an effective improvement of the swelling resistance upon blending. Also, the reinforcement of that blend with PET fibers results in further decrease in swelling values, in motor oil and brake fluid.

Therefore, blending of CR and NBR improved the swelling resistance of CR in motor oil and improved the swelling resistance of NBR in brake fluid. Also, the reinforcement of 50/50 CR/NBR blend with PET fibers results in further improvement of swelling resistance, both in motor oil and brake fluid.

Effect of different vulcanizing systems on the characteristics of CR/NBR composites

Ethylenethiourea (ETU)/MgO (0.5/4 in phr) curing system is commonly used to cure CR, whereas MBT/S curing system (with two different ratios 1.5/1.5 or 1/2, in phr), CBS/S (1/2, in phr) and TMTD (3, phr) curing systems are generally used to cure diene rubbers.

In the present investigation two groups of curing systems were examined to cure CR/NBR (50/50 by weight). Group 1 consists of half the amounts, mentioned above, of ETU/MgO combined with half amounts of MBT/S (with two different ratios), CBS/S or TMTD. Group 2 consists of the conventional amounts, mentioned above, of MBT/S (with two different ratios), CBS/S or TMTD, in absence of ETU/MgO.

The base recipe contains: CR 50, NBR 50, stearic acid 2 phr, ZnO 5 phr, IPPD 1 phr, HAF 40 phr, processing oil 5 phr, Hisil 5 phr, resorcinol 5 phr, HMT 3.2

phr and PET fibers (38mm length) 20 phr. Rheometric characteristics of the mixes and physicomechanical properties of the composites were determined and listed in Table 2.

Table 2 Formulations, rheometeric characteristics and physico-mechanical properties of the composites containing various vulcanizing systems

	Group t				Group 2			
Curing systems	315	M6	M7	M8_	M9	M10	M11	M12
MgO	2	2	2	2				
ETU	0.25	0.25	0.25	0.25	-		_	
MBT	0.75	0.50	_	_	1.50	1.00		
CBS		_	0.5				1.0	
TMTD				1.5		-	-	3.0
S	0.75	1.00	1.00		1.50	2.00	2.00	
Rheometric Characteristi	ics at 1:	52 ± 1°C	7					
Minimum torque (ML), dN.m.	13.0	9.5	10.0	11.0	10.0	11.0	11.0	10.5
Maximum torque (Mg), dN.m.	89	96	96	50	124	140	147	53
Cure time (tcso), min.	22	29.0	36.5	28.0	12.5	19.5	26.0	27.5
Scorch time (ts2), min.	1.25	1.50	1.75	2.00	1.00	1.25	1.75	2.25
Cure rate index (CRI), min. 1	4.8	3.6	2.9	3.8	8.7	5.5	4.1	4.0
Mechanical Properties at	25 °C							
Tensile strength, MPs (L)	10.5	11.6	11.5	5.4	11.2	11.6	12.0	5.0
Censile strength, MPa (T)	8.8	9.0	8.1	3.2	10.3	9.5	11.0	2.6
Elongation at break, % (L)	215	200	215	110	140	130	80	120
Elongation at break, % (T)	240	210	200	120	155	155	140	125
100% modulus, MPa (L)	7.6	7.2	6.7	4.7	10.7	9.9		4.8
100% modulus, MPa (T)	5.1	4.72	4.7	2.8	7.9	6.3	8.2	2.6
Young's modulus, MPs (L)	4.9	5.6	5.3	5.0	8.1	8.9	10.0	4.0
Young's modulus, MPa (T)	4	4.3	4.1	2.7	6.6	6.1	8.0	2.1
Swelling in toluene, at 25	°C .for	48 hrs						
Weight swell, %	123	114	113.5	122	105	96	92	118
Mechanical Properties at	ter the	rmal ae	cine at 9	0°C. fo	r 7 day			
Tensile strength, MPa (L)	10.4	11.1	11.2	5.1	11.6	11.1	11.9	5.1
Elongation at break, % (L)	175	165	170	105	115	100	55	110
100% modulus, MPa (L)	8.7	8.4	7.7	5	11.2	11.8	_	5.4
Young's modulus, MPa (L)	6	6.9	6.6	4.9	11.3	10.9	20.5	4.7
Swelling in oils at 100°C,	for 7 d	avs						
Weight swell, % in motor oil	5.4	5.9	4.9	6.2	4.4	4.6	4.4	5.6
Weight swell, % in brake fluid	17.6	16.8	16.5	18.6	15.8	16	16	18

⁽L) = Longitudinal direction(T) = Transverse direction

Table 2 shows that the use of the conventional systems (group 2) leads to better mechanical properties than those obtained by using group1 curing systems. Also, the use of MBT/S with two different ratios in both groups shows similar mechanical properties. On the other hand TMTD cured vulcanizate possesses the lowest tensile strength and modulus. However, CBS/S cured vulcanizate possesses the highest

maximum torque, tensile strength and modulus, but the shortest elongation at break and the least swelling value in toluene.

Mechanical properties of the vulcanizates were determined in L direction after thermal ageing for 7 days, as shown in Table 2. It is clear that tensile strength, elongation at break, 100% modulus and Young's modulus of all composites are almost unchanged upon ageing. Aged 100% modulus of CBS/S (1/2, in phr) vulcanizate is undetermined because its elongation at break is less than 100%.

The vulcanizates were exposed to swelling in motor oil and in brake fluid for 7 days, at 100°C. It is obvious from Table 2 that the group 2 vulcanizates possess lower swelling values than group 1 vulcanizates. Also, TMTD vulcanizates show higher swelling values than CBS/S or MBT/S vulcanizates in each group.

Effect of blend ratio on the properties of PET-CR/NBR composites

CR/NBR blends were prepared with various blend ratios. The formulations, rheometric characteristics of the mixes and pysico-mechanical properties of the composites are listed in Table 3. The base recipe contains: stearic acid 2 phr, ZnO 5 phr, IPPD 1 phr, HAF 40 phr, processing oil 5 phr, hisil 5 phr, resorcinol 5 phr, HMT 3.2 phr, PET fibers (38 mm length) 20 phr, CBS 1 phr and S 2 phr.

It is clear from Table 3 that the cure times of CR and NBR are approximately similar. The maximum torque decreases, while scorch time increases, by increasing NBR content in CR/NBR blend composites. Also, CR/NBR blend composites possess significantly shorter cure times and higher cure rate indecies, than those of the individual rubber composites.

Table 3 Formulations, rheometeric characteristics and physico-mechanical properties of the composites with various blend ratios

Elastomers	M13	M14	M11	M15	M16	
CR	100	75	50	25	0	
NBR	0	25	50	75	100	
Rheometric Characteristics	at 152 ± 1 °C					
Minimum torque (M _L), dN.m.	9.5	10	11	10.25	8	
Maximum torque (Mg), dN.m.	156	155	147	110	90	
Cure time (tcm), min.	35	30	26	24	37	
Scorch time (ts2), min.	1.00	1.50	1.75	2.25	2,75	
Cure rate Index (CRI), min."	2.94	3.5	4.1	5.00	2.92	
Mechanical properties at 25	•°C					
Tensile strength, MPa (L)	9.1	11.5	12.0	11.8	13.2	
Tensile strength, MPa (T)	8.3	10.2	11.0	11.3	10.9	
Elongation at break, % (L)	105	95	80	175	260	
Elongation at break, % (T)	135	140	140	190	290	
100% modules, MPa (L)	9.1			9.0	7.0	
100% modulus, MPa (T)	6.3	7.9	8.2	6.8	4.4	
Young's modulus, MPs (L)	8.7	9.7	10.0	6.7	4.7	
Young's modulus, MPa (T)	6.1	7.4	8.0	5.9	3.8	
Swelling in toluene, at 25°C	for 48 hrs					
Weight swelling , %	96	96.5	92	108	119	

⁽L) = Longitudinal direction (T) = Transverse direction

The obtained data show that the physico-mechanical properties are slightly improved with the increases of NBR content up to the ratio 50:50 then a decrease of the properties is obtained with further increase of NBR content.

The composites were exposed to thermal ageing for periods up to 7 days. The mechanical properties were measured in L direction. Figures 6-8 show that tensile

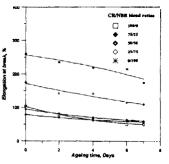


Figure 7 Elongation at break in L direction vs. ageing time, at 90 C, of the composites (20 phr PET) with various CR/NBR blend ratios.

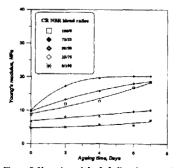


Figure 8 Young's modulus in L direction vs. agoing time at 90 C of the composites (20 phr PET) with various CR/NBR blend ratios.

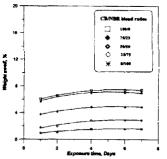


Figure 9 Swelling in motor oil vs. exposure time, at 100 C, of the composites (20 phr PET) with various blend ratios.

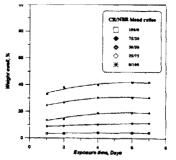


Figure 10 Swelling in brake fluid vs. exposure time, at 100 C, of the composites (20 phr PEI) with various blend ratios.

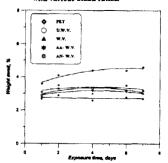


Figure 12 Swelling of the composites, containing, different fibers, in motor oil vs. exposure time, at 100 C.

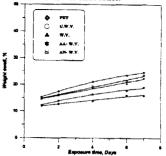


Figure 13 Swelling of the composites centaining, different fibers, in brake fluid vs. exposure time at 100 C.

strength of the composites with different blend ratios is almost unchanged upon ageing. Elongation at break slightly decreases, while Young;s modulus increases, upon ageing.

Of all CR/NBR blend ratios investigated, the 50/50 blend possesses high tensile strength, highest Young's modulus, but shortest elongation at breck, after thermal ageing. This makes that blend work at high stress condition with low strain.

Figures 9 and 10 show that the degree of swelling values of the composites in motor oil and in brake fluid slightly increase with the exposure time till 4 days, then remain constant with increasing the exposure time, at 100°C. It is obvious that CR composite possesses the highest swelling values in motor oil, while NBR composite possesses the highest swelling values in brake fluid. But CR/NBR (50/50) composite possesses medium swelling values, either in motor oil or in brake fluid. Thus, CR/NBR (50/50) composite can be used in both motor oil and brake fluid.

In summation, the composite containing CR/NBR (50/50 by weight) blend possesses good mechanical properties together with high thermal stability. Also, that composite has good swelling behaviour in motor oil, brake fluid and toluene.

Effect of grafted viscose fiber on CR/NBR vulcanizates

For further improvement of the swelling resistance of CR/NBR vulcanizates, viscose fibers were compared with polyester fibers and trials were made to increase the efficiency of viscose fiber via grafting with acrylic monomers. For this purpose, viscose short fibers were washed, firstly, in hot 0.1N solution of NaOH to remove any fatty materials on fiber surface, then dried to a constant weight. The washed viscose fibers (w.v.) were grafted with two different vinyl monomers, namely, acrylic acid (AA) and acrylonitrile (AN) using ceric ammonium nitrate as a redox initiator. The homo-polymer was extracted by acetone/ cyclohexanone mixture (50:50 by volume). The grafting yield was determined by the increase in weight and confirmed via IR spectroscopy (Figure 11a-c). Unwashed (u.w.v.), washed (w.v.) and grafted washed viscose fibers (AA-w.v. and AN-w.v.) were incorporated into rubber formulations in amounts equivalent to 20 phr fiber and compared with that containing 20 phr PET fiber (of the same length and denier), as shown in Table 4.

The base recipe contains: CR 50, NBR 50, stearic acid 2 phr, ZnO 5 phr, IPPD 1 phr, HAF 40 phr, processing oil 5 phr, hisil 5 phr, resorcinol 5 phr HMT 3.2 phr, CBS 1 phr and S 2 phr.

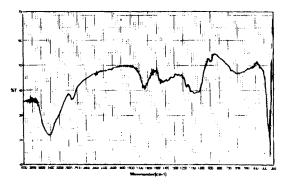


Figure 11a: IR Spectrum of ungrafted viscose fibers

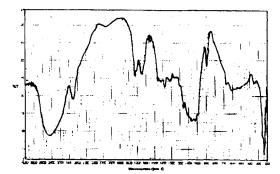


Figure 11b : IR Spectrum of AA grafted viscose fibers. Characteristic splitted band of carboxylic group at 1718 and 1645 cm⁻¹.

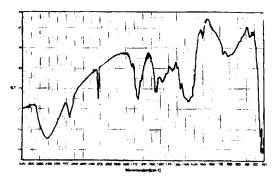


Figure 11c: IR Spectrum of AN grafted viscose fibers.

Characteristic band of cyano group at 2243 cm⁻¹.

Table 4 Formulations, rheometeric characteristics and physico-mechanical properties of the composites with grafted and ungrafted viscose fibers

Fiber	M11	M17	M18	M19	M20
PET fiber	20				
Unwashed viscoe fiber (u.w.v.)		20			_
Washed viscoe fiber (.w.v.)	_	_	20		
AA- grafted washed viscoe fiber (AAw.v.)			_	21.6	
AN- grafted washed viscoe fiber (ANw.v.)	_				45.6
Rheometric Characteristics at 152 ±	t 1°C				
Minimum torque (ML), dN.m.	10	7	7	8	9
Maximum torque (Mg), dN.m.	146	124	122	146	158
Cure time (tem), min.	29	24	23.5	25	20.6
Scorch time (ts2), mia.	2	1.5	1.5	1.75	1.5
Cure rate index (CRI), min. 1	3.7	4.4	4.5	4.3	5.3
Mechanical Properties at 25 °C					
Tensile strength, MPa (L)	12	12.9	11.6	11.3	10.1
Elongation at break, % (L)	80	100	75	130	45
100% modulus, MPa (L)	-	12.9		10.5	_
Young's modulus, MPa (L)	15	12.9	15.7	8.4	23.5
Swelling in toluene for 48 hrs, at 25'	'C				
Weight swell, %	92	72	69	80	63
Mechanical Properties after therma	l ageing	for 7 days.	at 90°C		
Tensile strength, MPa (L)	11.9	13.5	13	13.3	10.3
Elongation at break, % (L)	55	45	40	55	35
Young's modulus, MPs (L)	20.5	30	32.5	23.6	30_3

AA= Acrylic acid monomer, AN= Acrylonitrile monomer

From Table 4, it is clear that the removal of fatty materials from the viscose fiber surface by washing, improves the adhesion between fiber and rubber as shown from the decrease of both elongation at break and swelling in toluene and the increase in Young;s modulus. Also, acrylic acid grafted viscoes showed un remarkable effect, while, the acrylonitrile grafted viscose showed the lowest elongation at break, the lowest swelling in toluene and the highest Young's modulus.

From Table 4, it is obvious that all the investigated composites showed good thermal stability in the tensile strength after ageing for 7 days, while a slight decrease in the elongation at break was observed, upon ageing. On the other hand, acrylonitrile grafted viscose fiber- composite showed the lowest swelling values in motor oil and showed a comparable swelling values with PET fiber- composite in brake fluid, as shown in Figures 12 and 13. Therefore, swelling resistance of viscose fiber- composite is improved via grafting with acrylonitrile monomer.

CONCLUSIONS

- 1. A balance of properties can be obtained upon blending CR and NBR. Also, CR/NBR blend possesses suitable swelling values both in motor oil and brake fluid.
- Reinforcement of CR/NBR blend with PET fibers improves thermal stability and swelling resistance of the vulcanizate, in motor oil and in brake fluid.

- The composites cured with MBT/S, CBS/S or TMTD, in absence of ETU/MgO, show better performance against thermal ageing as well as better swelling resistance, than the composites cured in presence of ETU/MgO
- CBS/S (1/2, in phr) cured composite possesses the best mechanical properties, thermal stability and swelling resistance in toluene, motor oil and brake fluid.
- 5. Of all CR/NBR blend ratios investigated, the 50/50 blend possesses the best mechanical properties and swelling resistance in toluene together with high thermal stability.
- 6. Washed viscose fiber- composite showed comparable mechanical properties with PET fiber- composite. While acrylonitrile grafted viscose fiber leads to the lowest elongation at break, highest Young's modulus and good swelling resistance in both motor oil and brake fluid.

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