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Utilisation of IIR Tube Reclaim for Developing NR/IIR Blends

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Butyl (IIR) tube reclaim (RR) was mixed with carbon black filled natural rubber (NR) compounds at various percentages. The blend containing a low percentage of RR was found to show improved ageing resistance and improved processability with out much reduction in the mechanical properties.

KEY WORDS Isobutylene-isoprene rubber, natural rubber, reclaim

INTRODUCTION

Reclaim is the type of degraded rubber with unique properties and it is prepared from waste or worn-out rubber products. Reclaiming process imparts the necessary degree of plasticity to vulcanised rubber and thereby enabling it to be blended with natural rubber or synthetic rubber. Utilisation of waste is almost as old as rubber manufacturing since it offers processing as well as economic advantages.¹ The utilisation of scrap materials is an important factor in the expansion of the raw materials basis of Industry, the reduction of demand for primary raw materials and economisation of financial resources.

In the last few years considerable work has been done on reuse of scrap polymeric materials.^{2–5} Acetta and Vergnaud^{6,7} have studied vulcanisation of the rubber powder with vulcanising agents under normal conditions of temperature, time and pressure without adding fresh raw rubber. Phadke, Bhattacharya, Chakraborty and De^{8,9} have studied vulcanisation of reclaimed rubber and its blends with NR. Studies of Kretic¹⁰ and *et al.* showed that there is a significant drop in tensile strength even at low levels of scrap rubber.

In the present paper we report the effect of addition of Isobutylene-isoprene rubber (IIR) tube reclaim (RR) on the mechanical and rheological behaviour of natural rubber (NR).

EXPERIMENTAL

Materials

Natural Rubber: ISNR-5, Mooney viscosity ML(1 + 4) 100°C 85.3 (Rubber Research Institute of India)

Reclaimed Rubber: Butyl reclaim supplied by Elgi Rubber Products Ltd.

Rubber Additives: Zinc oxide, Stearic acid, Dibenzthiazyl disulphide (MBTS), Tetramethyl thiuram disulphide (TMTD), Sulphur, Carbon black (HAF N 330) and Naphthenic Oil used were rubber grade.

Natural rubber compounds containing various levels of butyl reclaim were prepared on a laboratory mixing mill (6 × 12") according to ASTM D 3182 (1982) as per formulation given in Table I. Cure characteristics of these compounds were determined using a Monsanto Rheometer R-100. Vulcanization was carried out in an electrically heated laboratory hydraulic press at 150°C. Dumbell shaped tensile test specimens were punched out of these compression moulded sheets along the mill grain direction. The tensile properties were then measured on a Zwick Universal Testing Machine model 1445 using a crosshead speed of 500 mm/min as per ASTM D412-80. Angular test pieces were punched out of the compression moulded sheets and tear resistance was measured on a Zwick UTM according to ASTM D-624. Samples for abrasion resistance, compression set and hardness were moulded and tested as per relevant ASTM standards.

TABLE I
Formulations

Mix	A	B	C	D	E	F
NR	100	90	80	70	60	50
RR	--	20	40	60	80	100
ZnO	5.0	5.0	5.0	5.0	5.0	5.0
Stearic acid	2.0	2.0	2.0	2.0	2.0	2.0
MBTS	0.6	0.6	0.6	0.6	0.6	0.6
TMTD	0.2	0.2	0.2	0.2	0.2	0.2
Sulphur	2.5	2.5	2.5	2.5	2.5	2.5
Carbon black (HAF N-330)	40	40	40	40	40	40
Naphthenic oil	8.0	8.0	8.0	8.0	8.0	8.0

TABLE II
Cure characteristics of the blends

Mix	Percentage of IIR reclaim					
	0	10	20	30	40	50
Scorch time, min	2.25	2.25	2.00	2.00	1.75	1.70
Optimum cure time, min	5.00	5.00	4.75	4.75	4.00	3.50
Cure rate (d.Nm/min)	18.6	21.8	23.3	25.2	27.0	28.7
Maximum torque, d.Nm	56	58	60	62	63	65
Reversion, number of units dropped in 5min.	2.5	2.0	1.75	1.50	0.75	Nil

The ageing resistance of the vulcanizates was studied after ageing the samples at 100°C for 24 hours in a laboratory air oven.

Processability of these compounds was then evaluated using a Brabender Plasticorder at different temperatures (80°C, 100°C and 120°C) and at different shear rates. (20, 40 and 60 rpms).

RESULTS AND DISCUSSION

The cure characteristics calculated from the cure curves are shown in Table II. The cure characteristics of the blends indicate that an increase in reclaimed rubber content decreases the scorch time, decreases the optimum cure time, increases the cure rate and reduces the reversion. The reduced scorch time, cure time and higher rate of cure may be due to the increased availability of curatives in the NR phase. As IIR is highly resistant to heat ageing the addition of RR to NR is found to improve the reversion resistance of NR. Maximum torque which is a measure of the modulus increases with the addition of reclaimed rubber.

Figure 1 shows the tensile and tear strength of the blends. There is a significant drop in tensile and tear strength even at low level of RR. Elongation at break and modulus at 100% elongation of the blends are shown in Figure 2. The increase in modulus at 100% elongation and reduction of the elongation at break of the vulcanizates with the addition of RR may be due to increased crosslink density in the NR phase due to the increasing amount of curatives in the NR phase. Figure 3 shows the retention in tensile strength and modulus at 100% elongation after ageing at 100°C for 24 hours with the level of RR. NR has got very poor ageing resistance as is evidenced from the figure.

But there is a drastic change in retention in tensile strength and also in modulus at 100% elongation on increasing the level of RR. As the percentage of RR in NR compound increases, it makes the vulcanizates more and more stiffer, which results in an increase in hardness and a decrease in abrasion resistance (Figure 4). Figure

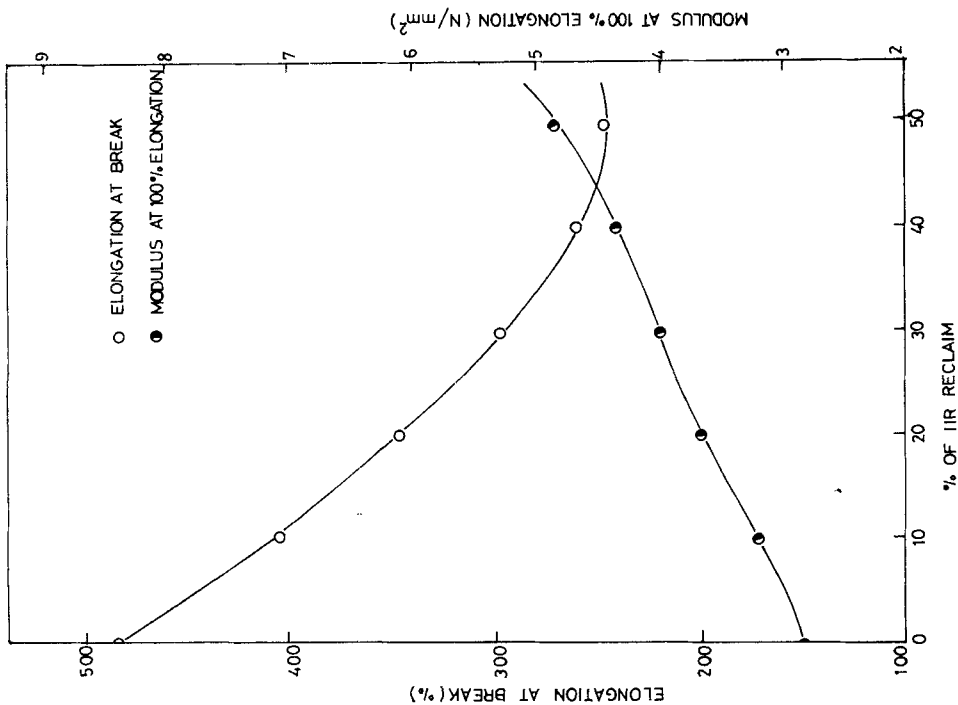


FIGURE 2. Variation of elongation at break and modulus with percentage of IIR reclaim.

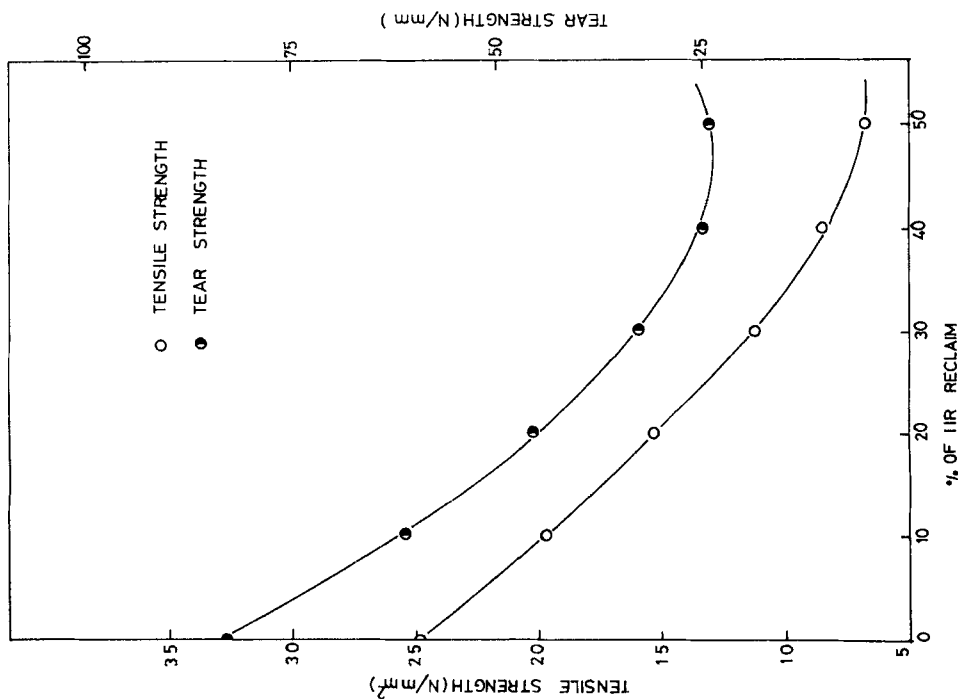


FIGURE 1. Variation of tensile strength and tear strength with percentage of IIR reclaim.

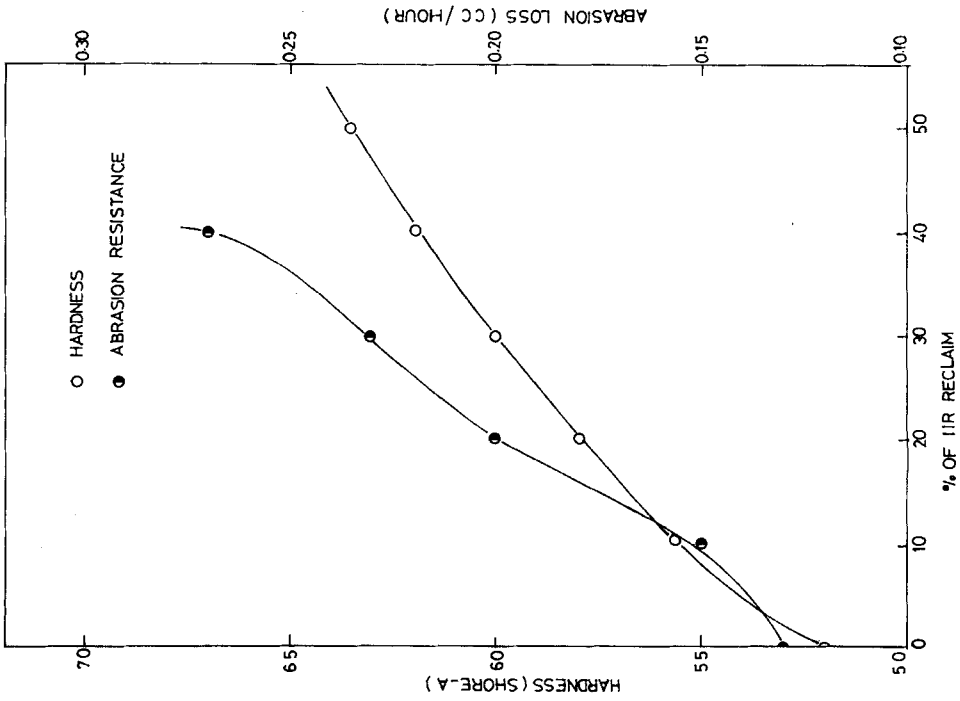


FIGURE 4 Variation of hardness and abrasion loss with percentage of IIR reclaim.

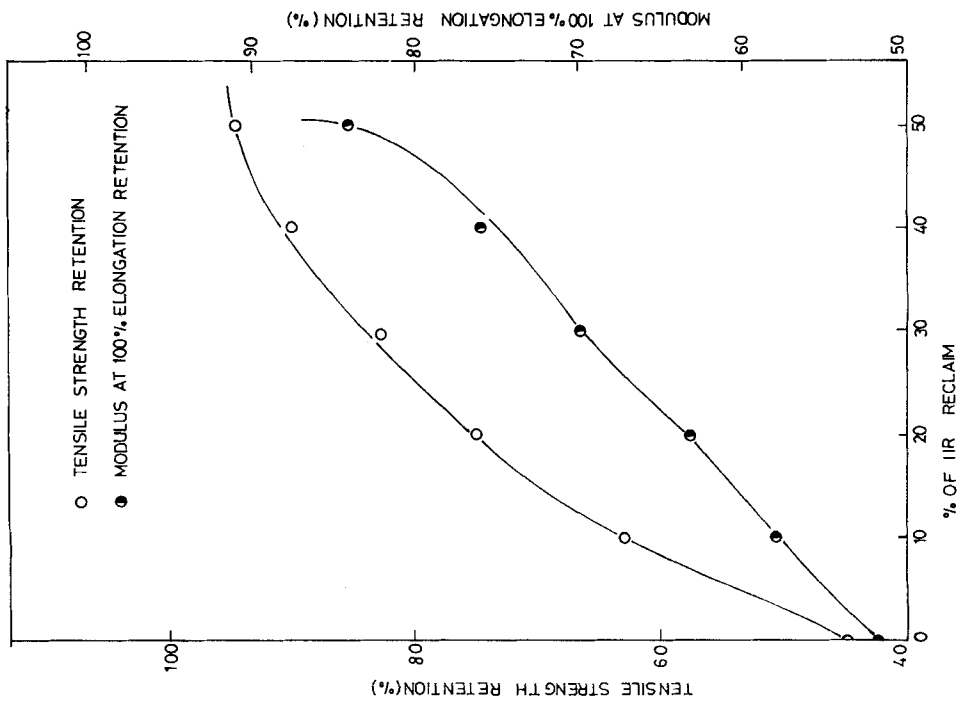


FIGURE 3 Variation of tensile strength retention and modulus retention with percentage of IIR reclaim.

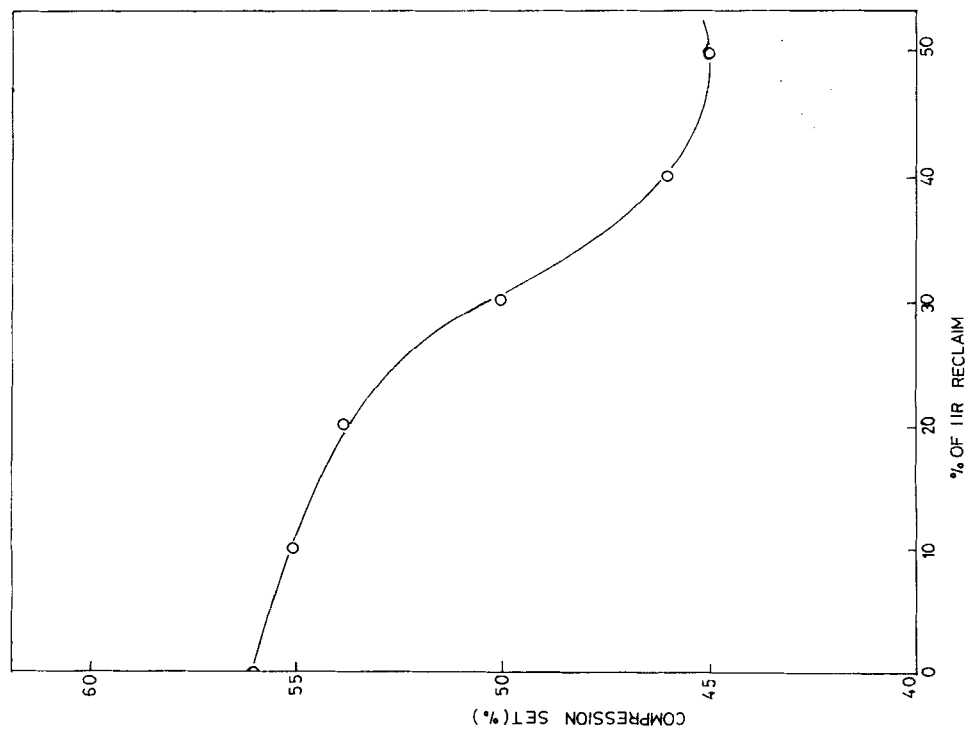


FIGURE 5 Variation in compression set with percentage of IIR reclaim.

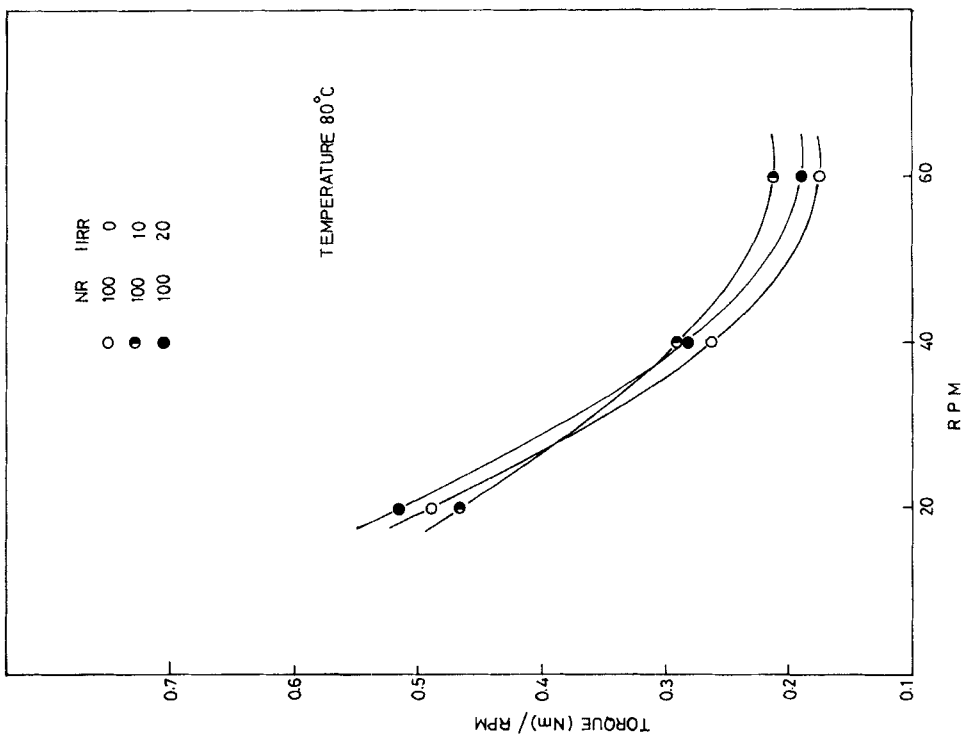


FIGURE 6 Variation of viscosity (Torque/rpm) with rpm for NR-IIR reclaim blends at 80°C.

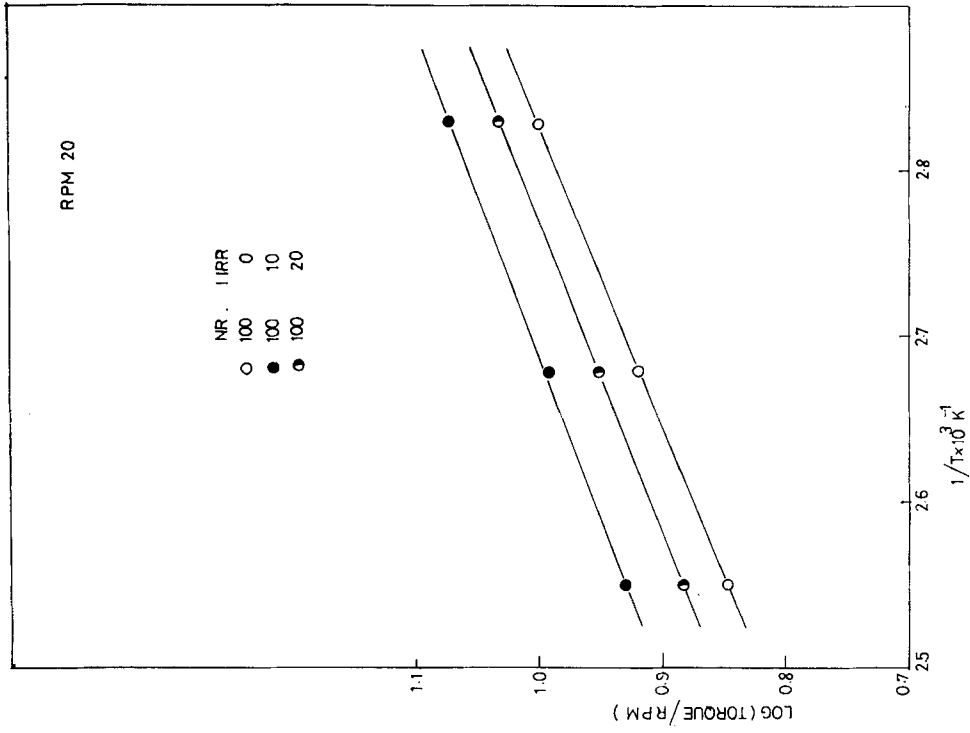


FIGURE 8 Variation of Viscosity (Torque/rpm) with temperature for NR/IIR reclaim blends at 20 r.p.m.

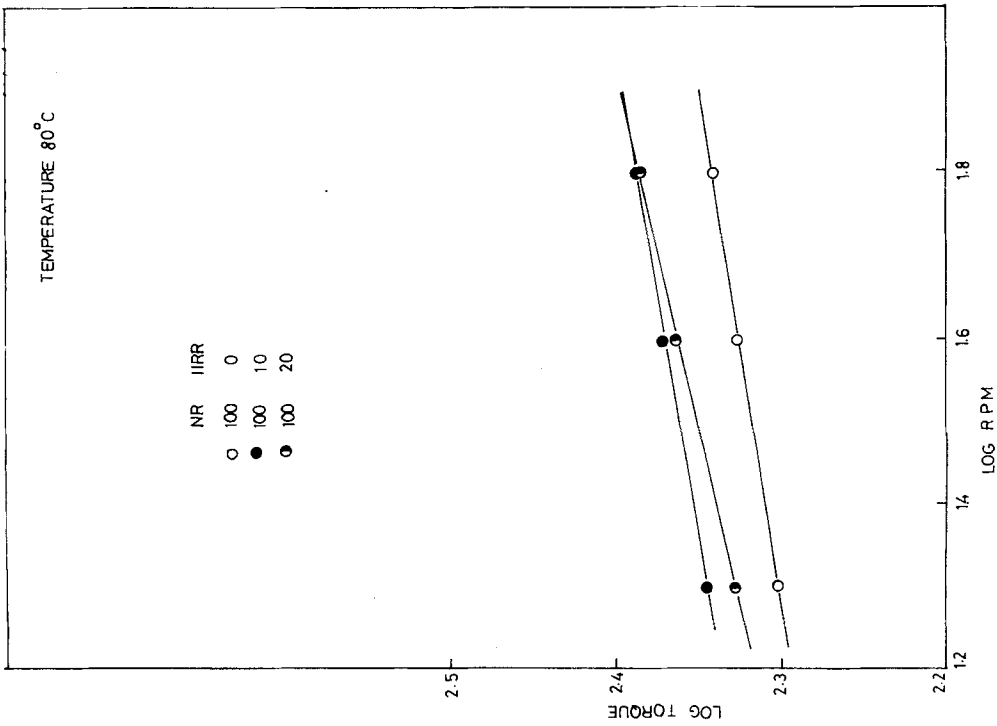


FIGURE 7 Variation of Torque with rpm at 80°C.

TABLE III
Power law indices for the blends

Percentage of IIR reclaim.	Power law indices at		
	80°C	100°C	120°C
0	0.07	0.09	0.125
10	0.08	0.09	0.19
20	0.125	0.22	0.25

TABLE IV
Activation energy of viscous flow for the blends

Percentage of IIR reclaim	Activation energy(kJ/mole) at		
	20 rpm	40 rpm	60 rpm
0	4.74	2.76	1.58
10	4.16	2.21	1.11
20	4.07	2.21	1.10

5 shows the compression set of the blends. The compression set value lies in between that of NR and IIR.

From the mechanical properties studied it seems that the best combination of service properties is reached at a content of 10–20% reclaimed rubber. So further processability studies were done with 10 and 20% reclaimed rubber.

Figure 6 illustrates the effect of viscosity of the blends with rpm (revolutions per minute) at a temperature of 80°C. The effect of rpm on viscosity is substantial as seen from the figures. Further, the addition of RR does not affect the viscosity of NR compounds.

Figure 7 shows the variation of torque with rpm. The power law indices were calculated from the equation $M = CS^a$ where M is the torque, S the rpm and C and a are constants, ' a ' represent the power law index.^{11,12} The extent of non-Newtonian behaviour of polymeric materials can be understood from the value of ' a '. Pseudoplastic materials are characterised by ' a ' below 1. Therefore a high value of ' a ' shows a low pseudoplastic or non-Newtonian nature of the material. The dependence of pseudoplasticity of the NR compound on temperature and loading of RR can be understood from the values given in Table III.

The values of ' a ' increases with increase in temperature and with the levels of addition of RR. This shows the decreased pseudoplasticity of NR/RR blends compared to the NR compound.

Torque/rpm can represent viscosity and the activation energy can be measured from torque rheometer data,³ if log torque/rpm is plotted against $1/T$. Figure 8 shows the effect of viscosity on temperature at 20 revolutions per minute. The activation energies of flow calculated from the slope of these lines are given in Table IV. The activation energy of a material provide valuable information on the sensitivity of the material towards the change in temperature. The higher the activation energy, the more temperature sensitive the material will be. From the table it is clear that NR has a higher activation energy compared to NR-RR blend. By the addition of 10% RR the activation energy for viscous flow is reduced to some extent. But further addition does not make any significant change.

CONCLUSIONS

The IIR reclaim possesses the parent polymer properties like good ozone and ageing resistance. There is a general reduction in physical properties with the addition of RR to NR compound. The addition of 10% by weight of RR in NR compound improves the processability and ageing resistance without much reduction in mechanical properties. The addition of RR does not require any special processing techniques, mixing cycles or equipments.

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