Chapter 4

Results and Discussion

4.1 Results and Discussion

In designing of compound for resin soles, there are three main factors considered in this work.

I Processability characteristics of the rubber compounds
II Paint coating characteristic of the vulcanized resin- rubber sheets
III Physical properties of the vulcanisates of the rubber compounds

Processing characteristics and their limits

Processing characteristics of any designed formulation should be satisfied with the following working limits to agree with the existing manufacturing processes.

Working limits of rheological properties for extrusion

Scorch Time (Sec)- TS-2.	70 – 100
Time to 90 % Cure (Sec) -TC-90	130 - 170
Minimum Torque (kgf-cm) - ML	10.00 – 12.00
Maximum Torque (kgf-cm) -MH	30.00 – 35.00

These limits are very important in compound mixing in internal mixer, extrusion process and compression moulding for resin rubber curing. On the other hand, final physical properties also depend on these values. In order to control above-mentioned working limits, it is essential to closely monitor the compound mixing process. It was found that the dumping temperature at mixing stage is the critical parameter, which should be closely monitored. In this project, it is understood that the dumping temperature of mixed compound lump at mixing stage should be between 135 C° and 145 C° to get proper processing characteristics.

When sulphur was added directly in the internal mixer, scorch time (TS 2) values were in lower side and minimum torque (ML) values were on higher side due to high temperature build up and the results caused mainly scorch and heat-build problem during extrusion process.

It has been found that instead of adding sulphur in the internal mixer, the sulphur mixing on a two roll can overcome these problems, because mill mixing is safe. It increases the Scorch time (TS 2) and reduces the temperature and viscosity of mixing Scorch time (TS 2) values were in lower side and Minimum torque (ML) values were on higher side, which affected the smooth flow of compound in the extrusion process. So adding of sulphur on the mill is more suitable to avoid any processing difficulty in extrusion process. Experimental results of the compound based on reclaimed rubber are given in the Table 4.1

Table 4.1 Rheometer data for Formulation 1.1-Reclaimed rubber/SBR (15:55) blend and Formulation 1.2-Reclaimed rubber/SBR (40:30) blend

Parameter	Formulation 1.1	Formulation 1.2
Scorch Time (Sec)	66	87
Time to 90 % Cure (Sec)	129	152
Minimum Torque (kgf-cm)	16.71	11.37
Maximum Torque (kgf-cm)	43.70	34.64

Initially 15 phr of reclaimed rubber was added with 55 phr SBR and Stearic acid 2 phr was introduced instead of Aktiplast PP for acceleration system since stearic acid is available at low cost and Rhenosin C 90 chemical was removed from the formulations. Processability was very poor in compounding as well as extrusion processes. So in order to increase safety period of compound (Scorch time TS 2) CBS was increased and PVI 0.3 phr was added to compound formulation 1.2, since CBS acts as delayed accelerator and PVI prevents pre-vulcanising in extrusion. On the other hand, reclaimed rubber was increased to 40 phr and Rhenosin C 90 was included to formulation 1.2 to overcome these processing difficulties. Introduction of high amount of reclaimed rubber to compounding formulation has shown very good flow properties with improved processability, not only in compounding but also in extrusion and compression moulding processes. Compound cost of formulation 1.2 was reduced further by reducing expensive SBR and increasing amount of low cost reclaimed rubber. Rheometer data for experiments based on natural rubber (RSS) are given in the Table 4.2

Table 4.2 Rheometer data for Formulation 2.1-Natural rubber alone (100 %) and Formulation 2.2-Natural rubber/SBR (37.5:45.8) blend with Tyre buffing dust (12.5) and Cotton flock (16.7)

Parameter	**Formulation 2.1**	**Formulation 2.2**
Scorch Time (Sec)	60.5	67
Time to 90 % Cure (Sec)	95	139
Minimum Torque (kgf-cm)	12.45	14.83
Maximum Torque (kgf-cm)	38.65	39.75

All compound formulations with natural rubber-RSS were failed due to processing problems. Due to its high stereoregularity, natural rubber crystallises spontaneously when it is stretched and sometimes it may lead to processing problem especially on extrusion though a die. Rheological characteristics of compound of natural rubber are varying from batch to batch due to inconsistency in properties of raw natural rubber and molecular chain lengths are very large and cannot controlled like in SBR. On the other hand pre-mastication of natural rubber is required prior to start the compounding which is an additional process, not needed in the case of synthetic rubber. Experimental results of natural rubber SLR-20 based formulations are given in the Table 4.3 and Table 4.4

Table 4.3 Rheometer data for Formulation **3.1** –Compound formulated with SLR 20 alone (100%) and Formulation3.2 - Compound formulated with SLR 20 alone (100%) with PVI

Parameter	**Formulation 3.1**	**Formulation 3.2**
Scorch Time (Sec)	60.7	71
Time to 90 % Cure (Sec)	100	108
Minimum Torque (kgf-cm)	6.63	6.13
Maximum Torque (kgf-cm)	33.32	33.42

First trial was done by introducing 100% SLR 20 instead of SBR and it has been found that the safety period of compound was not enough for extrusion due to high vulcanising rate of natural rubber. But minimum torque of SLR 20 based compound was less compared with conventional grade RSS due to lower viscosity of SLR 20 grades. In order to improve safety period of compound, PVI was added to same formulation. But attempt was not successful due to processing problems. As a result of that next trials were done with blend of SLR20 and SBR and rheometer data of this compounds formulations is given in the Table 4.3.

Table 4.4 Rheometer data for formulation 3.3 - Compound formulated with SLR 20 / SBR (32.5:32.5) blend and formulation 3.4 - Compound formulated with SLR 20 / SBR (32.5:32.5) blend with PVI

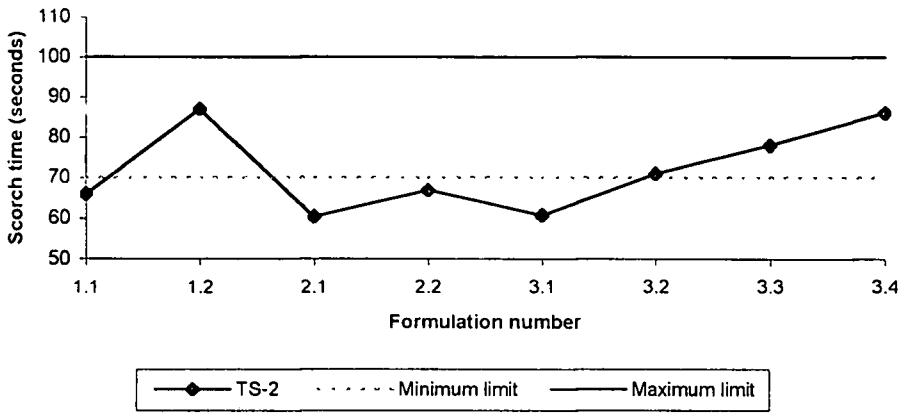
Parameter	**Formulation 3.3**	**Formulation 3.4**
Scorch Time (Sec)	78	86
Time to 90 % Cure (Sec)	120.8	137
Minimum Torque (kgf-cm)	7.42	7.97
Maximum Torque (kgf-cm)	32.94	31.66

In order to overcome processing problems and to improve safety period of the compound further, PVI was added to formulation based on blend of SLR 20/SBR. PVI is a prevulcanisation inhibitor for natural rubber. It is used with all sulfenamide accelerators to control processing safety with minimum changes in curing characteristics or vulcanisate properties. It improves green stock storage stability and can be used to recover scorch stocks. Useful in such applications as faster extrusion.

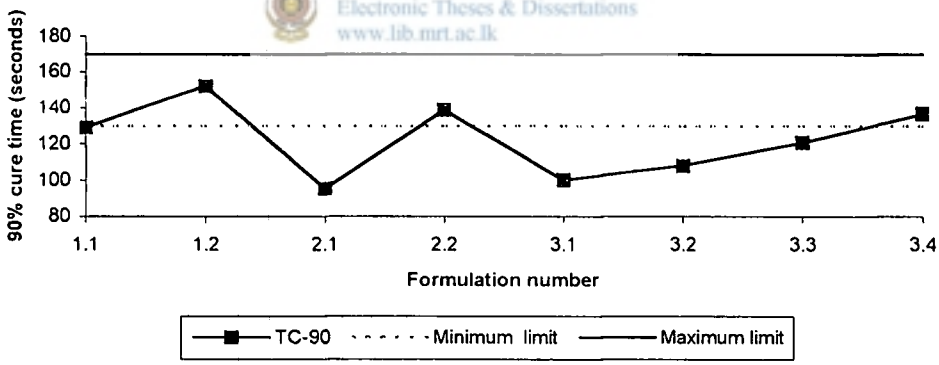
Comparison of flow properties of rubber compounds

a. Scorch Time (TS-2) and Time to 90% curing (TS – 90)

Comparision of TS-2 with working limit for defferent formulations



Comparision of TC-90 with working limits for different formulations



1.1 - Reclaimed Rubber / SBR (15:85) blend

1.2 - Reclaimed Rubber / SBR (40:30) blend

2.1 - Natural Rubber-RSS alone (100%)

2.2 - Natural rubber-RSS/SBR (37.5/48.8) blend

3.1 - Natural rubber- SLR 20 alone (100%)

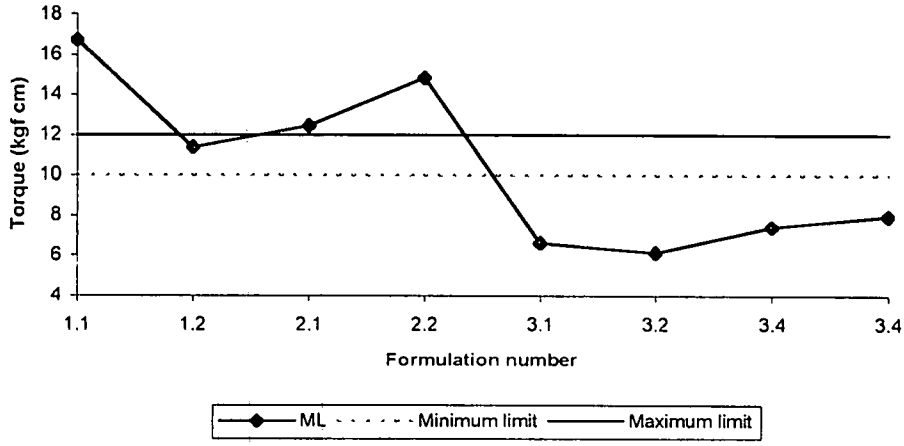
3.2 - SLR 20 alone (100%) with PVI

3.3 - Natural rubber- SLR 20 / SBR (32.5:32.5) blend

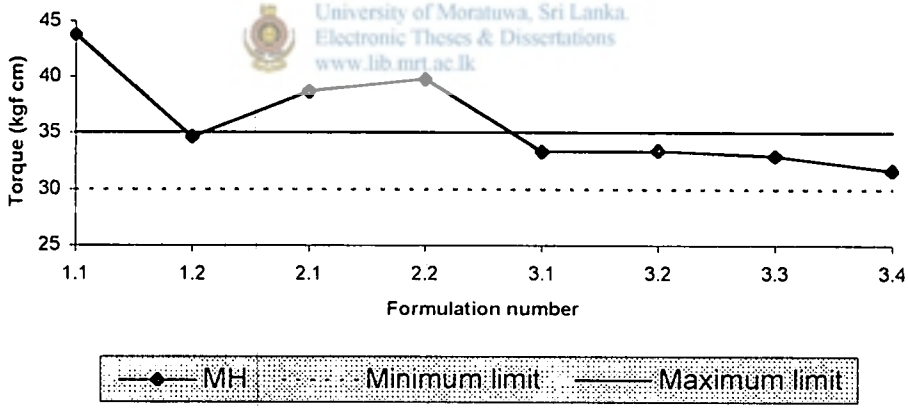
3.4 - Natural rubber- SLR 20 / SBR (32.5:32.5) blend with PVI

b. Minimum Torque (ML) and Maximum Torque (MH)

Comparision of ML with working limits for different formulations



Comparision of MH with working limits for different formulations



1.1 - Reclaimed Rubber / SBR (15:85) blend

1.2 - Reclaimed Rubber / SBR (40:30) blend

2.1 - Natural Rubber-RSS alone (100%)

2.2 - Natural rubber-RSS/SBR (37.5/48.8) blend

3.1 - Natural rubber- SLR 20 alone (100%)

3.2 - SLR 20 alone (100%) with PVI

3.3 - Natural rubber- SLR 20 / SBR (32.5:32.5) blend

3.4 - Natural rubber- SLR 20 / SBR (32.5:32.5) blend with PVI

The data of results indicate that the values obtained for flow properties (Scorch time, 90% curing time, Minimum torque and maximum torque) for various compounds formulations are not within the working limit as specified early, except formulations 1.2 (Compound formulated with Reclaimed Rubber / SBR (15:60) blend) and formulation 3.4 (Compound formulated with SLR 20 / SBR (32.5:32.5) blend with PVI)

II Paint coating characteristic of the vulcanised resin rubber sheets

Quality of the painted resin rubber sheet is generally investigated visually. But basically 3 tests, which were explained earlier, are carried out for painted sheets to check the paint coating strength of resin sheet to avoid paint stacking and paint peeling off when resin sheets are cleaned using solvent like acetone at soles assembling to shoe. Impurities present in Compound are seriously affecting the quality of the painted sheets. When supper shine colours are painted all compound impurities are appeared on the painted surface. So care must be taken to select suitable raw materials. Other factor, which affects the quality, is particle size of fillers of the compound formulation.

In formulation 2.2 introduced Buffing Dust and cotton flock were appeared on the surface of rubber sheets. As a result of that painted sheets were totally out of the required quality level.

Formulations 1.1, 2.1, 3.1, 3.2 and 3.3 were not tested for paint coating strength, since processing problem in extrusion. Of all these formulations, formulations 1.2 and 3.4 have shown promising results.

III Physical properties of vulcanised rubber sole sheet

Physical properties of the resin rubber sole required according to Shoes and Allied Trades Research Association (SATRA) are as follows

Hardness Shoe A	93 ± 2
Abrasion Resistance ($mm^{3)}$	≤ 400
Density (g/cm^3)	≤ 1.5

Physical properties of the compound formulations based on blend of reclaimed rubber and SBR are given in the Table 4.5

Table 4.5 Physical Properties

Parameter	**Formulation 1.1**	**Formulation 1.2**
Hardness (Shore A)	94	93
Abrasion resistance (mm^3)	379	348
Density (g/cm^3)	1.452	1.360

Vulcanisate of compound formulations 1.1 and 1.2 have shown promising results for physical properties, which recommended by SATRA.

Physical properties of the compound formulations based on natural rubber- RSS are given in the Table 4.6

Table 4.6 Physical Properties

Parameter	**Formulation 2.1**	**Formulation 2.2**
Hardness (Shore A)	87	85
Abrasion resistance (mm^3)	295	245
Density (g/cm^3)	1.480	1.248

The data of results of introduction of natural rubber (RSS) (Formulation 2.1 and 2.2) indicate that the value obtained for hardness of the cured sheet is not within the international requirement for resin rubber soles. Low hardness of natural rubber based compound formulations was due to reduction of amount of high styrene resin. Filler loading was increased to get required hardness, but attempt was not successful. Vulcanisate of compound formulation based on natural rubber has excellent abrasion resistance and it is very light due to low density.

Physical properties of compound formulations based on natural rubber -SLR 20 are given in the Table 4.7 and 4.8

Table 4.7 Physical Properties

Parameter	**Formulation 3.1**	**Formulation 3.2**
Hardness (Shore A)	92	92
Abrasion resistance (mm^3)	294	294
Density (g/cm^3)	1.348	1.348

Table 4.8 Physical Properties

Parameter	**Formulation 3.3**	**Formulation 3.4**
Hardness (Shore A)	92	92
Abrasion resistance (mm^3)	338	316
Density (g/cm^3)	1.360	1.369

Physical properties of the Formulations 3.1, 3.2, 3.3 and 3.4 are within the SATRA standards specified earlier.

4.2 Conclusion

Experimental results show that the compound based on natural rubber-RSS alone (Formulation 2.1) and compound containing RSS, tyre-buffing dust, cotton flock (formulation 2.2) was not suitable due high heat built up during extrusion process and vulcanisate showed low hardness in both formulations. Further buffing dust and cotton flock were appeared on the surface of rubber sheets, which were made from formulation 2.2. As a result of that cured sheets were also found to be below the level of standard quality.

Compound made with styrene butadiene rubber- Reclaimed rubber (formulation 1.2) was very successful due to better flow properties not only in compounding but also in extrusion and compression moulding processes.

Reclaimed rubber is low cost than SBR and it was found that buffing dust and kaolin in the amount 65 phr and 90 phr respectively were useful in the SBR/Reclaimed rubber blend, for the cost reduction. In this new formulation compound cost was reduced by 30% as a result of introduction of these local raw materials (Reclaimed rubber, Buffing dust and Kaolin).

In addition to the above formulation the formulation (3.4), based on peptised natural rubber SLR 20)/SBR blend (50:50) have also indicated results in compliance with the standard of international organization.

In view of these, formulations based on blend of reclaimed rubber/styrene butadiene rubber (Formulation 1.2) and formulation based on blend of Peptised natural rubber SLR 20/ styrene butadiene rubber (formulation 3.4) are found to be the most suitable formulation for production of resin-rubber soles in local rubber industry.

4.3 Further work

Further investigation should be made on designing rubber compounds with commonly available compounding ingredients such as fillers, softeners etc to reduce the cost.

A comprehensive investigation should also be necessary for the effect of reclaimed rubber on colour staining during service especially in styrene butadiene rubber/ reclaimed rubber blend based resin rubber soles.

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