Indian J. Nat. Rubb. Res. 2 (1) : 13–26, 1989

STUDIES ON THE USE OF 1, 2-POLYBUTADIENE IN MICROCELLULAR SOLES

Jacob K. Varkey, N. M. Mathew and P. P. De

Varkey, Jacob K., Mathew, N. M. and De, P. P. (1989). Studies on the use of 1,2-polybutadiene in microcellular soles. Indian J. Nat. Rubb. Res. 2(1) : 13–26.

The use of 1, 2-polybutadiene (1,2-PB) as a substitute for styrene-butadiene rubber (SBR)-high styrene resin in conventional microcellular (M.C) sole compounds was studied. Effects of concentration of 1,2-PB, filler combinations and concentration of blowing agent on technical properties of microcellular soles prepared from blends of natural rubber (NR) and 1,2-PB are discussed. Cell characteristics of seven selected vulcanizates were studied using a scanning electron microscope (SEM). It was found that blending of 1,2-PB with NR results in higher expansion, lightness, higher flex life and lower blooming in M. C soles. However, other technical properties are slightly inferior to those of M.C soles prepared using SBR-high styrene resin. A combination of china clay, precipitated silica and aluminium silicate in the ratio 60 : 30 : 15 is found to be an acceptable filler combination for NR/1,2-PB based M. C soles.

Key words—Natural Rubber, 1,2–Polybutadiene, Styrene-butadiene rubber, Blend, Microcellular sole.

Jacob K. Varkey (for correspondence) and N. M. Mathew, Rubber Research Institute of India, Kottayam-686 009, India and P. P. De, Rubber Technology Centre, Indian Institute of Technology, Kharagpur–721 302, India.

INTRODUCTION

Cellular soles are used in footwear mostly owing to their high strength to weight ratio. Conventionally a blend of natural rubber (NR) or styrene-butadiene rubber (SBR) and SBR - high styrene resin is used for making microcellular soles. Various types of polymers and their blends are used in footwear to achieve specific combination of properties such as lightness, wearing comfort, stiffness and durability. SBR-high styrene resins improve hardness, stiffness, abrasion resistance, etc. The acceptable range of properties required for rubber based M. C soles is specified in IS 10702–1985.

Supply of styrene for the production of SBR is reported to be decreasing (Elliot, 1974; Hall, 1974). Attempts have been going on to develop a substitute for SBR in general purpose applications, including microcellular soles, where use of styrene rich SBR is common. One of the most promising substitutes for SBR is 1,2-polybutadiene which possesses an overall balance of properties for many general purpose applications (Railsback and Stumpe, 1976). It exhibits thermoplastic elastomeric property due to stereoregular structure with a controlled degree of crystallinity. Resistance to abrasion, cut growth, flexing and ozone are reported to be better for 1, 2-PB compared to styrene - butadiene block copolymers and other polymers like ethylene vinyl acetate (Japan Synthetic Rubber Co., RB 810, RB 820).

The present work is an attempt to evaluate the suitability of 1, 2-PB as a substitute for SBR-high styrene resin in microcellular sole compounds.

EXPERIMENTAL

(a) MATERIALS

The following materials were used in the present investigations.

1. **Polymers:**

Natural rubber	ISNR–5
SBR - 1958	SBR-high styrene resin. Bound styrene content - 49%
SBR - 1502	Bound styrene content - 23%
1, 2-PB	JSR RB 820, 1, 2 content - 92%, Crystallinity - 25%, Specific gravity - 0.902

ISNR - 5 was obtained from the Pilot Crumb Rubber Factory, Rubber Board, Kottayam. SBR- 1958 and SBR- 1502 were manufactured by M/s Synthetics and Chemicals Limited, Bareilly. JSR RB 820 was manufactured by M/s Japan Synthetic Rubber Company Limited.

2. **Fillers:**

Precipitated silica : Specific gravity-1.95

China clay : Specific gravity-2.6

Aluminium silicate : Specific gravity-2.0

Microcrumb : Particle size-400-800μm

3. **Curatives:**

MBTS (Dibenzthiazyl disulphide) and TMTD (Tetramethyl thiuram disulphide)

4. **Blowing agent:**

Dinitrosopentamethylene tetramine (DNPT), 40% active, commercial grade

5. **Other chemicals:**

Zinc oxide, stearic acid, titanium dioxide, diethylene glycol and sulphur, commercial grade

(b) PREPARATION OF COMPOUNDS

The compounds were prepared in a Shaw Intermix-MK-3, size KO, set at 40 rpm and at an initial temperature of 80°C. The mixing schedule was as follows.

0 min — NR

2 min — SBR/1,2-PB

4 min — Filler

8 min — Dump

The final mixing of curatives and blowing agent was done in a 15 cm x 30 cm laboratory mixing mill set at a friction ratio of 1 : 1.25 and a roll temperature of 80°C.

(c) MOULDING OF SHEETS

Microcellular sheets were moulded in a 45 cm x 45 cm hydraulic press having steam heated platens. The mould was loaded with three per cent excess of the mix on volume basis. The temperature and pressure of moulding were 150°C and 10.5 MPa respectively. The compound was pre-cured in the mould to 80 per cent of the maximum cure time, measured using a Monsanto rheometer - R 100. The expanded precured sheet was then post-cured in an oven for 3 hours at 80°C to complete the curing process.

(d) TESTING

The sheets were tested for technical properties such as relative density, hardness, change in hardness after ageing, split tear strength, compression set, water absorption, heat shrinkage, room temperature shrinkage and flex resistance as per IS-6664 - 1972, IS-3400 - 1974 and IS-10702 - 1985. Abrasion resistance was tested using DIN abrader as per DIN 53516 with a load of 5 N.

(e) CELL STRUCTURE

Observations were made using a JEOL - 35C model scanning electron microscope (SEM). Cell structure studies were done on a cut surface of the sample. Soon after cutting, the specimens were sputter coated with gold. Photomicrographs of the special features of each sample were also taken.

RESULTS AND DISCUSSION

(1) EFFECT OF CONCENTRATION OF 1, 2-PB ON TECHNICAL PROPERTIES OF SOLES

Recipes of the mixes are given in Table 1.1. M. C sheets prepared from compounds containing more than 60 parts of 1,2 - PB were defective, and hence studies were limited to sheets prepared with a maximum of 60 parts of 1,2-PB. Technical properties of the M.C sheets are given in Table 1.2.

The relative density decreased from 0.75 to 0.38, as the 1,2-PB content was increased from 0 to 60 parts. The size of the expanded sheet increased with increasing 1,2-PB content. The lowering of relative density, with increasing 1,2-PB content, is due to the lower density of this material (Japan Synthetic Rubber Company Limited, JSR RB 820) and the higher expansion it caused due to its thermoplastic nature (Kimura *et al*, 1975; Takeuchi *et al*, 1974).

Hardness and split tear strength of the product decreased with increasing 1,2-PB content. Kuriakose (1986) reported that the hardness of natural rubber/1,2-PB blend vulcanizates increased with increased 1,2-PB content. The observed decrease in hardness and split tear strength in the case of the cellular product is likely due to the higher expansion favoured by 1,2-PB which, being thermoplastic, remains less viscous at the moulding temperature. The variations in hardness and split tear strength are given in Figs. 1.1 and 1.2.

Compression set increased with increasing 1,2-PB content. It has already been reported (Kuriakose, 1986) that the extent of crosslinking decreased with increasing 1,2-PB content. The lowering of rheometric torque with increasing 1,2-PB content as reported in Table 2.2 also supports this view. Thus the higher compression set may be attributed to the thermoplasticity of the polymer, failure of the thinner microcellular walls resulting from higher expansion and the lower extent of crosslinking with increased 1,2-PB content.

Heat shrinkage increased with increasing 1,2-PB content. Since 1,2-PB has higher gas permeability (Lundberg, 1979) an increase in heat shrinkage is expected. Shrinkage at room temperature was found to be almost constant irrespective of the polymer variation. Water absorption, which is a measure of the open cells produced in the expanded sheet, increased with increasing 1,2-PB content. This again could be attributed to the higher expansion. As the cells expand and the microcellular walls become thinner, chances for breakage of cell walls, resulting in intercommunicating cells, are more.

Abrasion loss of the M.C sheet also increased with increase in 1,2-PB content. In solid vulcanizates of NR/1,2-PB blends, abrasion loss was reported diminishing with increasing 1,2-PB content (Kuriakose, 1986). In the case of M.C sheets, the observed increase in abrasion loss with increase in 1,2-PB content is due to the higher expansion resulting in thinner cell walls which favours easier wearing of the product. The variation of abrasion loss with polymer content is given in Fig. 1.3.

1, 2-PB based soles showed higher flex resistance, compared to SBR - high styrene based soles. NR and SBR based soles on storage showed blooming of chemicals and chemical residues, whereas 1, 2-PB based soles did not show any such tendency. Compared with SBR-high styrene resin based soles, 1, 2-PB based soles showed slightly inferior technical properties. This may be due to the slower cure rate and the lower degree of crosslinking of 1, 2-PB as well as the higher expansion of the sole.

Table 1.1. **Formulations of Mixes 1—8**

Materials	Mix number							
	1	2	3	4	5	6	7	8
Natural rubber	70	35	100	90	80	70	60	40
SBR — 1958	30	30	..	..	..	..	..	..
SBR — 1502	..	35	..	..	..	..	..	..
1,2-Polybutadiene	..	..	..	10	20	30	40	60
Stearic acid	2	2	2	2	2	2	2	2
Zinc oxide	5	5	5	5	5	5	5	5
Titanium dioxide	5	5	5	5	5	5	5	5
Antioxidant SP	1	1	1	1	1	1	1	1
Precipitated silica	30	30	30	30	30	30	30	30
China clay	60	60	60	60	60	60	60	60
Aluminium silicate	15	15	15	15	15	15	15	15
Diethylene glycol	2	2	2	2	2	2	2	2
Naphthenic oil	10	10	10	10	10	10	10	10
MBTS	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8
TMTD	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Sulphur	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6
DNPT	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0

Table 1.2. **Technical Properties of Mixes 1–8**

Property	Mix number							
	1	2	3	4	5	6	7	8
Maximum torque (dN.M)	51	57	60	53	46	45	40	30
Minimum torque (dN.M)	8	17	11	12	10	10	6	5
Time for optimum cure (min.)	4.7	4.5	3.4	3.2	3.4	4.5	4.9	6.4
Cure rate index	55.5	55.5	58.5	58.5	58.5	33.3	33.3	22.2
Relative density	0.502	0.54	0.75	0.71	0.65	0.48	0.45	0.38
Hardness (Shore A)	43	47	35	34	32	30	29	27
Change in hardness (After ageing at 100±1°C, 24 hrs)	1	1	-1	0	0	2	2	0
Compression set (%)	20.0	19.0	8.2	8.8	12.5	24.7	29.5	34.5
Split tear strength (N)	36.0	54.0	49.5	48.0	45.0	35.0	33.0	32.0
Expansion ratio*	1.7	1.327	1.326	1.348	1.398	1.77	1.795	2.179
Abrasion loss (mm^3)	320	255	158	199	214	337	358	434
Water absorption (%)	12.5	16.7	7.2	9.5	11.0	34.5	33.5	38.3
Shrinkage at 100±1°C(1 hr)(%)	1.2	0.65	0.65	0.91	0.99	1.76	1.98	3.1
Room temperature shrinkage (%)	0.33	0	0.34	0.32	0.33	0.33	0.33	0.33
Flex resistance : Kilocycles to crack initiation	300	500	>800	>800	>800	>800	>800	>800

$$\text{*Expansion ratio} = \frac{\text{Area of the M. C sheet}}{\text{Area of the mould}}$$

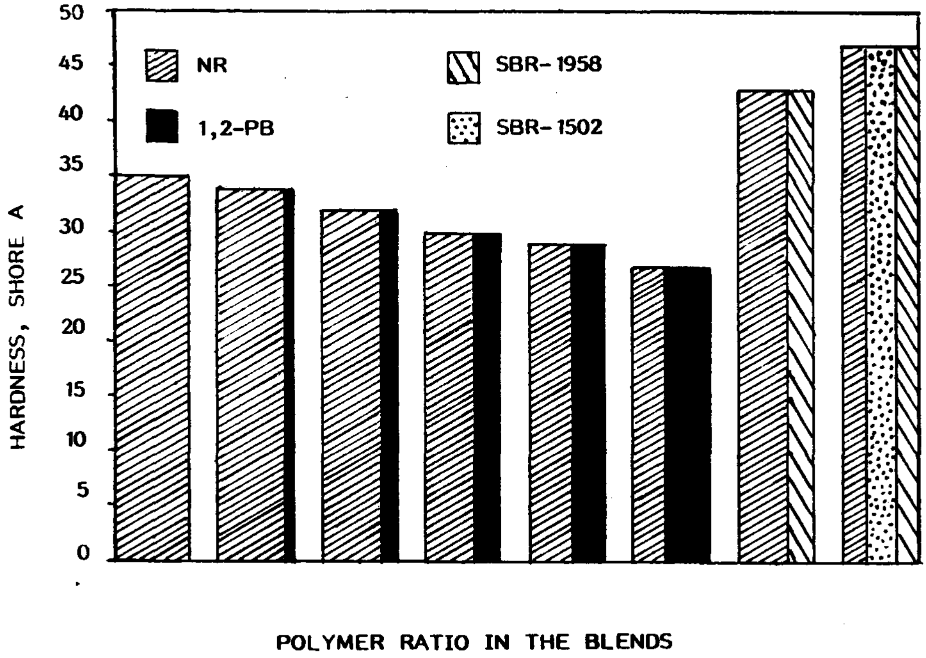

Fig. 1.1 Changes in hardness with polymer variation

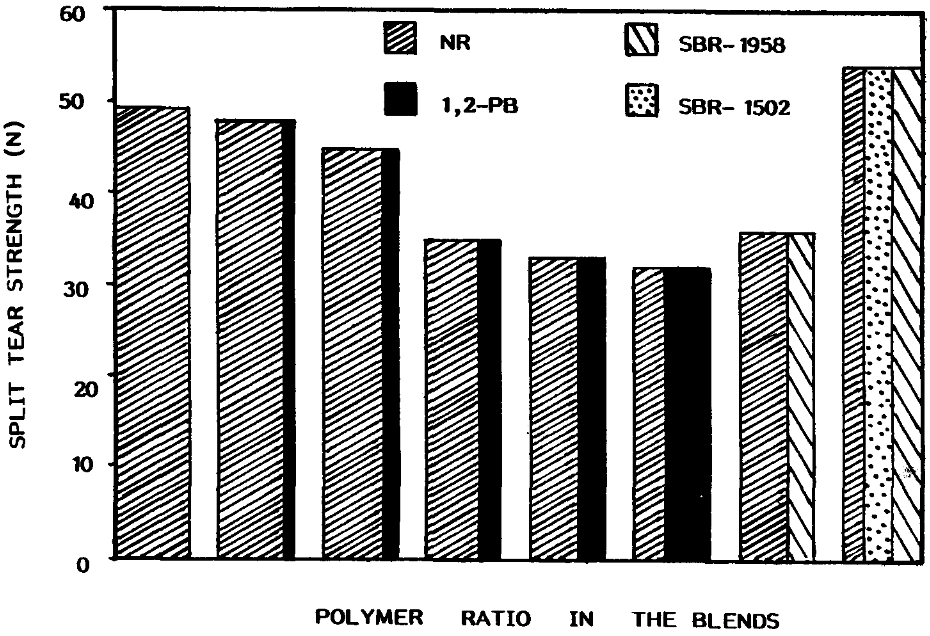

Fig. 1.2 Changes in split tear strength with polymer variation

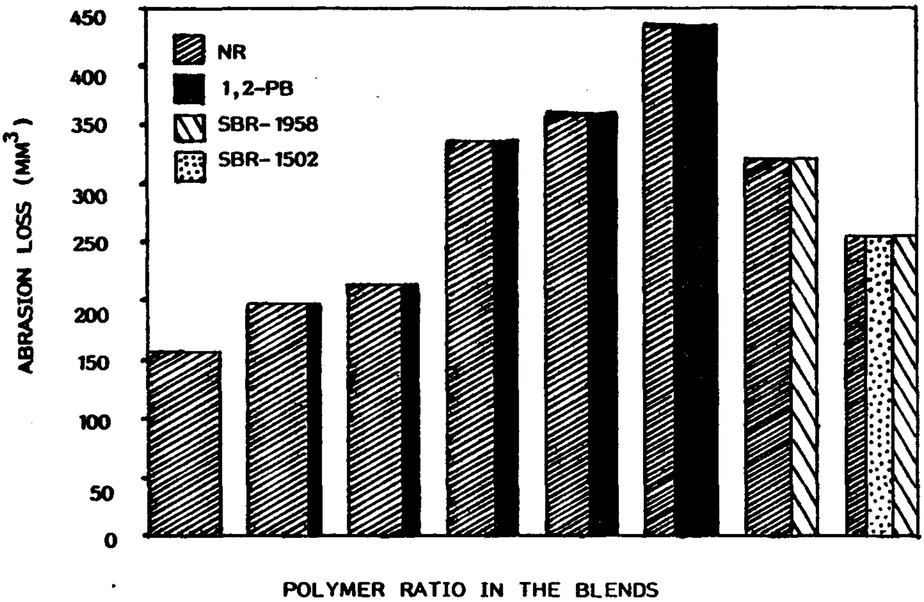

Fig. 1.3 Changes in abrasion loss with polymer variation

(2) Effect of filler

To study the effect of varying proportions of commonly used fillers such as china clay, precipitated silica, aluminium silicate and microcrumb in 1, 2-PB based M.C soles, seven compounds were prepared. The formulations of the mixes and the technical properties of the sheets prepared therefrom are given in Tables 2.1 and 2.2, respectively.

Substitution of china clay with precipitated silica, aluminium silicate and microcrumb reduced the relative density of the product. It is seen from Table 2.2 that as china clay was replaced by precipitated silica, the viscosity of the mix in terms of the minimum torque decreased, which accounts for the higher expansion. Also, it was reported that silica-filled 1, 2-PB compounds showed poor polymer-filler interaction (Bhagawan, 1987). Thus the lower specific gravity of the filler and the increased expansion, caused by lower viscosity and reduced polymer-filler interaction may be the reasons for the observed decrease in relative density of the product.

Hardness and split tear strength of the product were slightly reduced with increased substitution of china clay with precipitated silica and aluminium silicate. Though this filler alteration is expected to give better reinforcing properties in NR, the observed decrease in properties may be due to the poor polymer-filler interaction in the 1, 2-PB phase. The microcrumb-filled product also showed lower hardness and a slightly lower split tear strength compared to the china clay-filled one.

Increased silica/aluminium silicate/microcrumb loaded product showed higher compression set. This is a consequence of the higher expansion resulting in thin walls, which fails under compressive load leading to a higher set value. The observed increase in water absorption and abrasion loss of the product (Fig. 2) with progressive increase in silica, aluminium silicate and microcrumb also is a consequence of the increased expansion resulting from the filler alteration.

Microcrumb-filled soles showed poor flex resistance. Compared to the other fillers, the non-homogeneity and the larger size of the microcrumb are the probable causes for the quicker flex failure. It is already known that the size of filler particle is an important factor in determining the flex fatigue life of rubber products (Hess, 1965).

Table 2.1. **Formulations of Mixes 9–15**

Material	Mix number						
	9	10	11	12	13	14	15
Natural rubber	70	70	70	70	70	70	70
1,2-polybutadiene	30	30	30	30	30	30	30
Stearic acid	2	2	2	2	2	2	2
Zinc oxide	5	5	5	5	5	5	5
Titanium dioxide	5	5	5	5	5	5	5
Antioxidant SP	1	1	1	1	1	1	1
Precipitated silica	30	40	40	40	40	40	40
China clay	60	50	40	30	20	10	0
Aluminium silicate	15	15	25	35	35	35	35
Microcrumb	..	..	..	..	10	20	30
Diethylene glycol	2	2	2	2	2	2	2
Naphthenic oil	10	10	10	10	10	10	10
MBTS	0.8	0.8	0.8	0.8	0.8	0.8	0.8
TMTD	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Sulphur	1.6	1.6	1.6	1.6	1.6	1.6	1.6
DNPT	7.0	7.0	7.0	7.0	7.0	7.0	7.0

Table 2.2. **Technical Properties of Mixes 9–15**

Property	Mix number						
	9	10	11	12	13	14	15
Maximum torque (dN.M)	45.0	42.5	42.5	39	38	38.5	37.5
Minimum torque (dN.M)	10	7.0	7.0	5.5	6.0	6.5	6.5
Time for optimum cure (min)	4.5	5.6	5.5	6.1	5.4	4.9	4.5
Cure rate index	33.3	28.5	30.3	24.4	27.7	34.4	39
Relative density	0.48	0.405	0.413	0.403	0.399	0.399	0.374
Hardness (Shore A)	30	27	27	27	27	28	28
Change in hardness (After ageing at 100±1°C, 24 hrs)	2	4	2	3	3	4	2
Compression set (%)	24.7	44	45.5	45.5	47.0	42.0	45.0
Split tear strength (N)	35.0	35.0	32.5	32.5	33.5	33.5	33.5
Expansion ratio	1.769	1.91	1.965	1.939	1.939	1.918	1.965
Abrasion loss (mm^3)	337	520	484	545	651	585	577
Water absorption (%)	34.5	41.6	42.7	46.7	46.4	45.9	48.1
Shrinkage at 100±1°C (1 hr)(%)	1.76	0.67	1.68	1.33	1.307	0.977	0.66
Room temperature shrinkage (%)	0.33	0.35	0.36	0.36	0.36	0.37	0.34
Flex resistance : Kilocycles to crack initiation	>500	>500	>500	>500	170	200	170

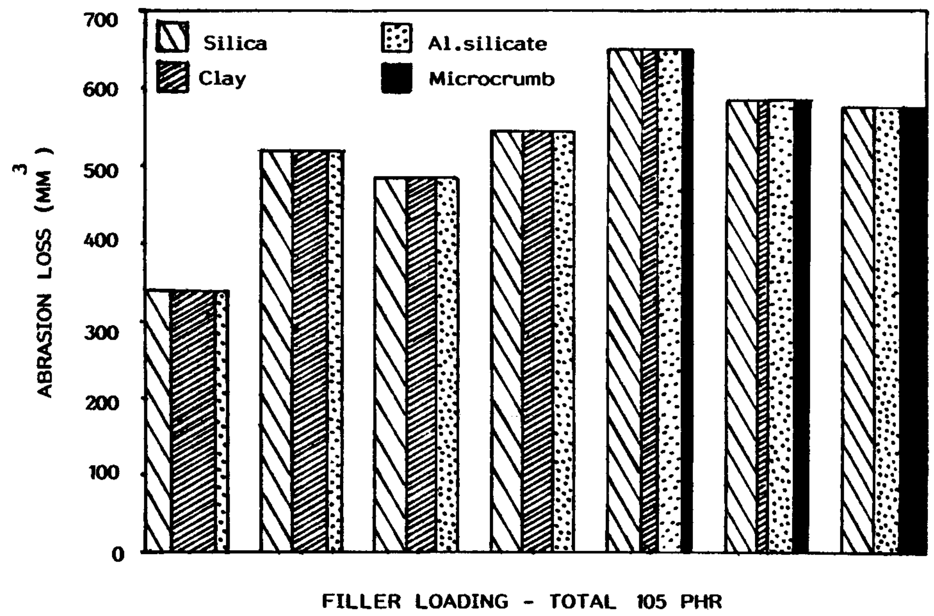

Fig. 2. Changes in abrasion loss with filler variation

(3) EFFECT OF CONCENTRATION OF THE BLOWING AGENT

Different levels of the blowing agent, DNPT, were studied for compounds with three different filler loadings. The formulations used and the technical properties are given in Tables 3.1 and 3.2, respectively.

Relative density, hardness and split tear strength decreased with an increase in the concentration of blowing agent. A progressive increase in abrasion loss was also observed with an increase in the blowing agent concentration (Fig. 3). The decrease in split tear strength and the increase in abrasion loss are consequences of the thinner cell walls, resulting from higher expansion. This view is also supported by the SEM photomicrographs.

Though increased blowing contributes to a higher output, it adversely affects some of the critical properties like hardness, split tear strength and abrasion loss.

Table 3.1. **Formulations of Mixes 16–24**

Materials	Mix number								
	16	17	18	19	20	21	22	23	24
Natural rubber	70	70	70	70	70	70	70	70	70
1,2-polybutadiene	30	30	30	30	30	30	30	30	30
Stearic acid	2	2	2	2	2	2	2	2	2
Zinc oxide	5	5	5	5	5	5	5	5	5
Titanium dioxide	5	5	5	5	5	5	5	5	5
Antioxidant SP	1	1	1	1	1	1	1	1	1
Precipitated silica	30	30	30	30	40	40	40	40	40
China clay	60	60	60	60	40	40	40	40	40
Aluminium silicate	15	15	15	15	25	25	25	0	0
Diethylene glycol	2	2	2	2	2	2	2	2	2
Naphthenic oil	10	10	10	10	10	10	10	10	10
MBTS	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8
TMTD	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Sulphur	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6
DNPT	7.0	6.0	5.0	4.0	7.0	6.0	5.0	6.0	5.0

Table 3.2. **Technical Properties of Mixes 16–24**

Property	Mix number								
	16	17	18	19	20	21	22	23	24
Maximum torque (dN.M)	45	44	46.5	50	42.5	40.0	40.0	39.0	39.0
Minimum torque (dN.M)	10	6.0	6.0	6.0	7.0	6.0	6.5	7.0	7.0
Time for optimum cure (min)	4.5	4.0	4.2	4.7	5.1	6.1	6.7	7.0	6.5
Cure rate index	33.3	45.5	45.5	45.5	35.7	26.3	26.3	21.2	25.0
Relative density	0.48	0.524	0.58	0.77	0.41	0.468	0.54	0.46	0.58
Hardness (Shore A)	30	34	39	45	27	32	35	29	37
Change in hardness (After ageing at 100±1°C, 24 hrs)	2	4	5	6	2	3	6	4	1
Compression set (%)	24.7	20.0	8.7	7.3	45.5	28.6	20.0	28	10.7
Split tear strength (N)	35.0	40.5	50.0	57.5	32.5	34.0	40.0	40.0	46.0
Expansion ratio	1.769	1.608	1.469	1.284	1.965	1.706	1.577	1.713	1.506
Abrasion loss (mm^3)	337	319	267	206	484	432	384	432	286
Water absorption (%)	34.5	28.1	26.4	18.9	42.7	32.3	28.4	32.5	28.2
Shrinkage at 100±1°C (1 hr) (%)	1.76	1.2	1.2	1.0	1.6	1.7	1.5	2.6	1.6
Room temperature shrinkage (%)	0.33	0.28	0.22	0.2	0.36	0.3	0.24	0.35	0.32
Flex resistance :									
Kilocycles to crack initiation	>600	>600	>600	>600	>600	>600	>600	>600	>600

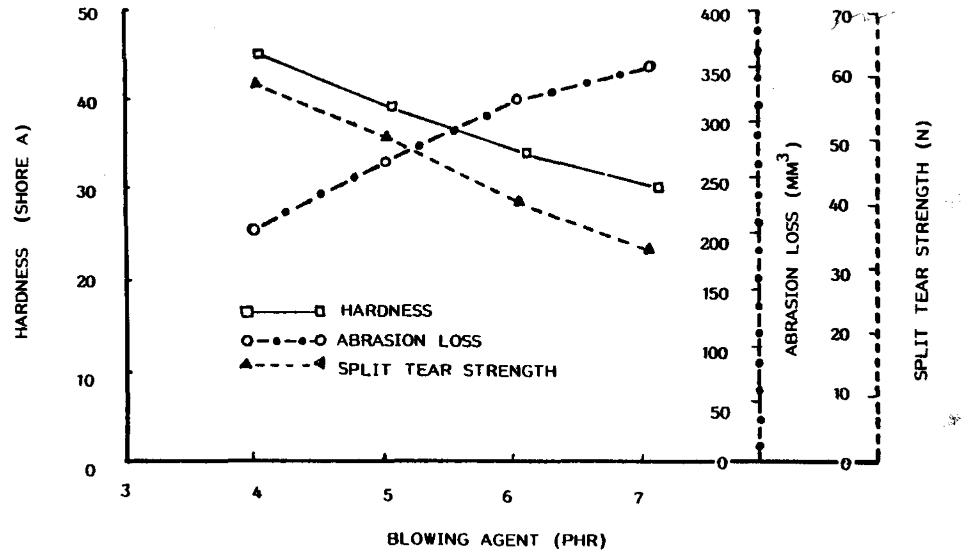

Fig. 3. Variation of hardness, abrasion loss and split tear strength with concentration of blowing agent (Mixes 16-19)

(4) CELL STRUCTURE

Observations with SEM were made only on seven selected vulcanizates (Table 4.1).

The product prepared from natural rubber alone (Mix 25) had comparatively smaller cells with appreciably thick walls (Fig. 4.1). The cells had an approximate diameter of 10 μm. The introduction of SBR - 1958 (Mix 26) caused a larger expansion of the microcellular sheet (Fig. 4.2). The average size was more and their walls were not as thick as in the case of pure natural rubber. The maximum diameter of the cell was around 100 μm. When 1, 2 - PB was blended with natural rubber instead of SBR-1958, as in Mix 27, the expansion was still larger as is evident from Fig. 4.3, the diameter often exceeding well over 100μm. The cell walls were much thinner compared with those observed in Mix 25.

A reduction in the level of the blowing agent reduced the expansion, which however did not cause a reduction in the cell size as is evident from Figs. 4.3 to 4.6 of the Mixes 27 to 30 containing seven, six, five and four phr of the blowing agent, respectively. The soles with 5 and 6 phr of blowing agent were found to give the largest cells. The cells in these vulcanizates were more circular in shape, with the cell walls becoming thicker as the proportion of the blowing agent was reduced.

When microcrumb was added as a filler (Mix 31) in place of china clay, a higher expansion was obtained (Fig. 4.7).

Table 4.1. **Formulation of Mixes for SEM Studies**

Material	Mix number						
	25	26	27	28	29	30	31
Natural rubber	100	70	70	70	70	70	70
SBR — 1958	..	30	..	..	..	..	..
1,2-polybutadiene	..	..	30	30	30	30	30
Stearic acid	2	2	2	2	2	2	2
Zinc oxide	5	5	5	5	5	5	5
Antioxidant SP	1	1	1	1	1	1	1
Titanium dioxide	5	5	5	5	5	5	5
Precipitated silica	30	30	30	30	30	30	40
China clay	60	60	60	60	60	60	0
Aluminium silicate	15	15	15	15	15	15	35
Microcrumb	..	..	..	..	..	..	30
Diethylene glycol	2	2	2	2	2	2	2
Naphthenic oil	10	10	10	10	10	10	10
MBTS	0.8	0.8	0.8	0.8	0.8	0.8	0.8
TMTD	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Sulphur	1.6	1.6	1.6	1.6	1.6	1.6	1.6
DNPT	7.0	7.0	7.0	6.0	5.0	4.0	7.0

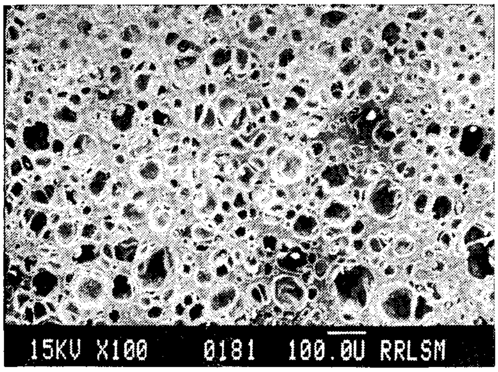

Fig. 4.1. SEM photomicrograph of expanded sole from Mix 25

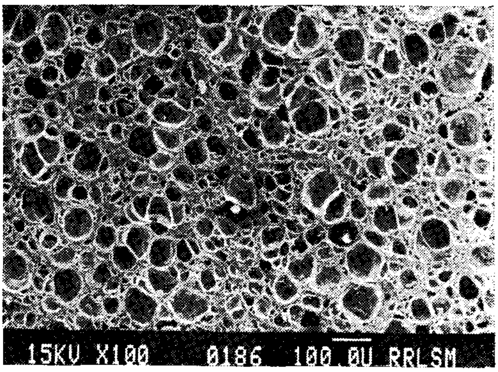

Fig. 4.2. SEM photomicrograph of expanded sole from Mix 26

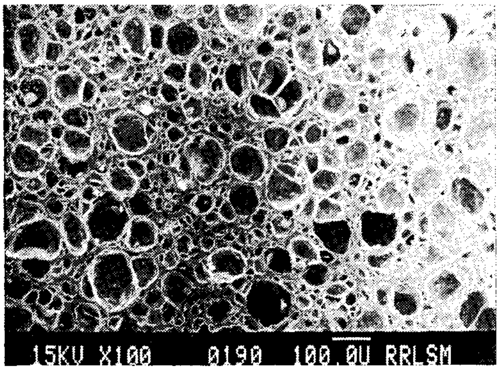

Fig. 4.3. SEM photomicrograph of expanded sole from Mix 27

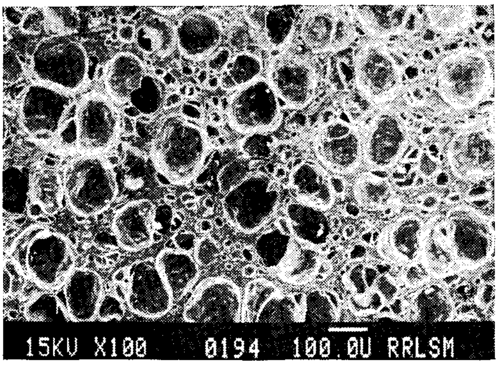

Fig. 4.4. SEM photomicrograph of expanded sole from Mix 28

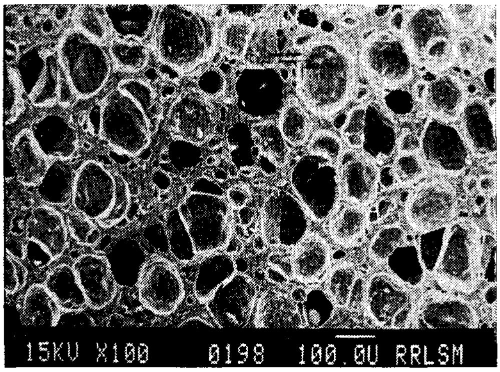

Fig. 4.5. SEM photomicrograph of expanded sole from Mix 29

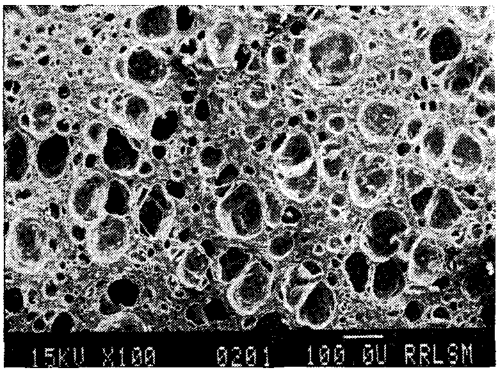

Fig 4.6. SEM photomicrograph of expanded sole from Mix 30

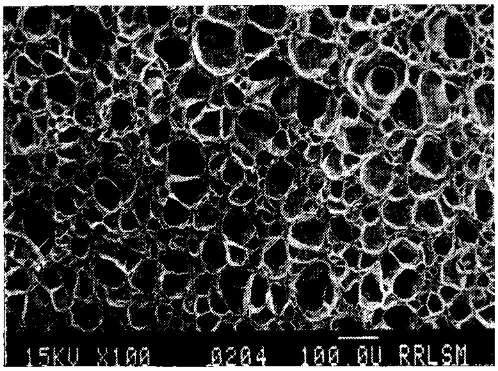

Fig. 4.7. SEM photomicrograph of expanded sole from Mix 31

CONCLUSIONS

In the manufacture of microcellular soles, blending of 1,2-polybutadiene with natural rubber can contribute to higher output, lightness, higher flex life and less blooming. Microcellular soles prepared from a 70/30 blend of natural rubber/1,2 - polybutadiene show slightly inferior technical properties compared to the SBR-high styrene resin based one. This can be attributed to the slow cure rate and the lower degree of crosslinking of 1, 2-PB compared to the styrene-butadiene rubber. A combination of china clay, silica and aluminium silicate in the proportion of 60:30:15, was found to be acceptable for NR/1,2-PB based soles. Of the different mixes studied, a 70:30 blend of NR/1, 2-PB with 7 phr of blowing agent and with the above filler combination gave the best possible set of properties required for the sole. Though a reduction in blowing reduced the output, it improved the technical properties of the product. Scanning electron microscopic studies served as a supporting evidence for the observed variation in properties of the different soles.

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