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RICE HULL ASH AS A FILLER IN MICROCELLULAR SOLES

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Fully burnt rice hull (rice hull ash) was tried as a low cost filler in place of precipitated silica in NBR/PVC based microcellular soles. The mechanical properties of the soles containing silica and ash are found to be comparable. The expansion is marginally higher in the presence of ash, which permits to reduce the amount of blowing agent. Cell structure of microcellular sheets remains unchanged when silica is replaced by ash.

Keywords: White ash; Silica; NBR/PVC blend; Microcellular soles; Mechanical properties

INTRODUCTION

Microcellular soling (MC soles) is the name applied to a range of expanded soling materials produced by the use of suitable compounds containing blowing agents. MC soles are used for making foot-wears due to their high strength to weight ratio, wearing comfort, stiffness and durability. Various types of polymers and their blends are used in MC soles to achieve specific combination of properties such as lightness, wearing comfort, stiffness and durability. Blends of NR/HSR, NR/EVA, NR/1,2-polybutadiene, NBR/PVC *etc.* are used for making MC soles. Fillers are used in MC soles for property improvement and cost reduction. Clay, precipitated silica, precipitated $CaCO_3$ *etc.* are commonly used as fillers in MC soles.

The production of rice, which is one of the major food crops of the world, generates major wastes like rice hull and straw. Disposal of rice

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hull is a serious problem and usually large amounts accumulate in the vicinity of rice mills. The rice hull is conventionally used as a fuel, but this generates another waste product, namely rice hull ash. Fully burnt rice hull is known as white ash and which contains about 85% silica [1]. As silica is commonly used as a filler in MC soles, white ash could be used as a potential low cost filler in place of silica. This study reports the use of fully burnt rice hull (white ash) as a substitute for precipitated silica in microcellular soles based on NBR/PVC blends.

EXPERIMENTAL

Materials Used

Nitrile Rubber (NBR): Aparene N-553 NS, bound ACN content 33% (supplied by Apar Polymer India Ltd.)

Polyvinyl chloride (PVC): Suspension polymer, K value 65.

Additives: Zinc oxide, stearic acid, zinc stearate, titanium dioxide, wood rosin, naphthenic oil, dioctyl phthalate, ethylene glycol, styrenated phenol, clay, precipitated silica and Azodicarbonamide (ADC) used were commercial grade.

Vulcafor F and tetramethyl thiuram disulphide (TMTD) were obtained from M/s. Bayer India Ltd.

Amine terminated liquid natural rubber (ATNR) was prepared in the laboratory by the procedure described elsewhere [2].

White Ash

Chemical analysis of the ash shows that it contains mostly acid soluble silicious material. It contains about 85% silica. The acid soluble portions contain some metal salts. Specific gravity of the ash used is 2.80.

Preparation of NBR/PVC Blend

Blends of NBR and suspension PVC were prepared in a Brabender Plasticorder model PL 3S using roller type mixing head set at 150°C and at a speed of 30 rpm. NBR was added, one minute was given for homogenizing and then PVC, premixed with stabilizer and plasticizer, was added [3]. The blending was completed in another 7 minutes. The formulation used is shown in Table I.

The NBR/PVC blend was compounded in a two roll mill (150 × 300 mm) at a friction ratio 1:1.25 according to the formulation given in Table II. The amount of white ash was varied from 30 to 60 phr. The amount of blowing agent (ADC) was gradually decreased from 9.75 to 9 phr with increase in the amount of white ash.

The cure characteristics of NBR/PVC blends were determined on a Goettfert Elastograph model 67.85.

Preparation of Microcellular Sheets

The NBR/PVC based MC sole compound was moulded in an electrically heated hydraulic press at 160°C. The mould was filled

TABLE I Formulation for PVC compound

Ingredients	*(Phr)*
Polyvinyl chloride	100
Zinc oxide	4
Magnesium oxide	4
Stearic acid	2
Dioctyl phthalate	40

TABLE II Formulation for NBR/PVC based MC sole compounds

Ingredients	1	2	3	4	5	6	7	8
NBR in (NBR/PVC blend)	100	100	100	100	100	100	100	100
Sulphur	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
ZnO	3	3	3	3	3	3	3	3
Stearic acid	1	1	1	1	1	1	1	1
Zinc stearate	1	1	1	1	1	1	1	1
Titanium dioxide	4	4	4	4	4	4	4	4
Silica	30	40	50	60	–	–	–	–
Clay	60	60	60	60	60	60	60	60
White ash	–	–	–	–	30	40	50	60
Diethylene glycol	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Amine terminated liquid natural rubber	5	5	5	5	5	5	5	5
Wood rosin	2	2	2	2	2	2	2	2
Vulcafor F	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
TMTD	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Azodicarbonamide	10	10	10	10	9.75	9.50	9.25	9.0

with 3 to 5 per cent excess of the compound on a volume basis. The compound was precured in the mould up to 70% of their optimum cure time. The expanded sheet was then post cured in an air oven for 2 hours at 70°C.

The mechanical properties such as relative density, hardness, compression set, water absorption and split tear strength were evaluated according to the relevant Indian Standards. Split tear strength was tested using a Zwick universal testing machine model 1445. Abrasion loss was tested using a DIN Abrader as per DIN 53516 with a load of 5 N.

The cell structure of the microcellular soles was studied using an optical microscope of magnification 33.

RESULTS AND DISCUSSION

Table III shows the cure characteristics of the compounds containing silica and white ash in NBR/PVC based MC sheets. There is a marginal decrease in cure time of the mixes containing white ash. This slight reduction in cure time may be due to the decreased adsorption of accelerator on the surface of white ash compared to silica.

Figure 1 shows the variation of expansion ratio and relative density with increase in the amount of filler loading in NBR/PVC based MC sheets. Expansion of MC sheets is found to be marginally lower when silica is replaced by white ash and hence relative density is marginally higher.

Figure 2 shows the variation of split tear strength and abrasion loss with increase in the amount of filler loading in NBR/PVC based MC sheets. Split tear strength marginally improves when silica is replaced by white ash. This is due to the reinforcing effect of white ash. This

TABLE III Cure characteristics of the compounds containing silica and white ash

Amount of silica (Phr)	*Cure time (min)*	*Amount of white ash (Phr)*	*Cure time (min)*
30	11	30	10.9
40	10.3	40	10.1
50	9.5	50	9.3
60	8.2	60	7.9

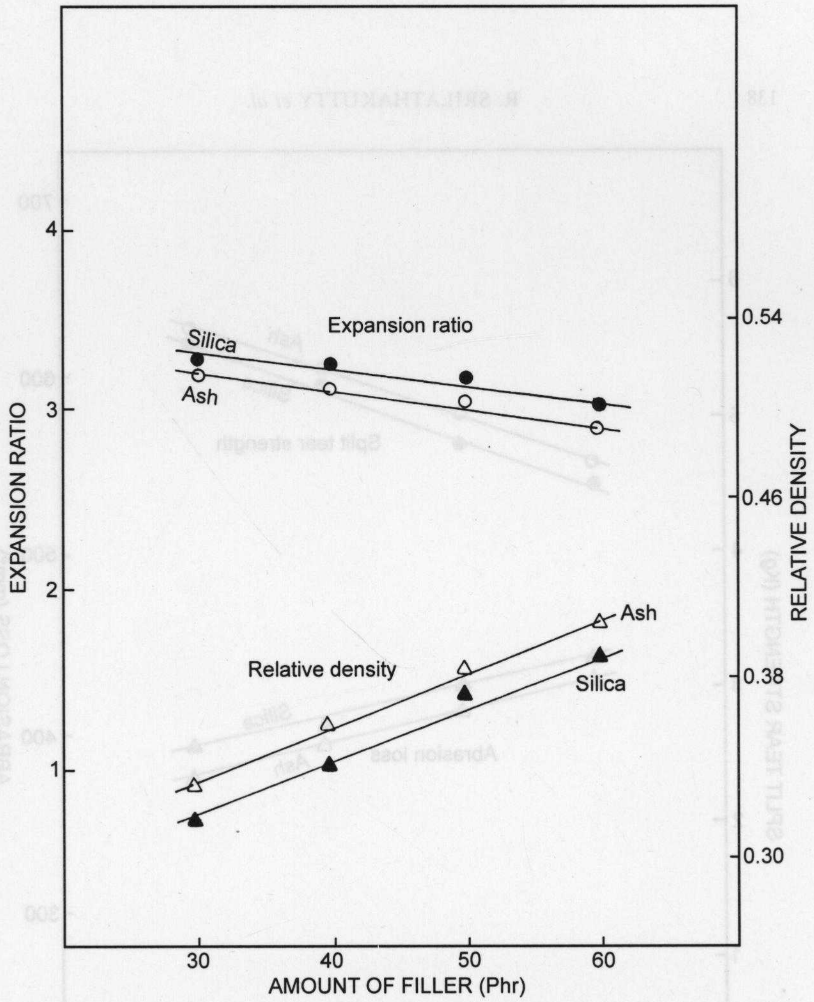

FIGURE 1 Variation of expansion ratio and relative density with increase in filler loading.

indicates that white ash can bring about equivalent reinforcement compared to that of silica. The abrasion loss of the MC sheets decreases when silica is replaced by white ash. This may due to the stronger cell walls resulting from the lower expansion [4].

Figure 3 shows the variation of hardness and heat shrinkage with increase in the amount of filler in NBR/PVC based MC soles.

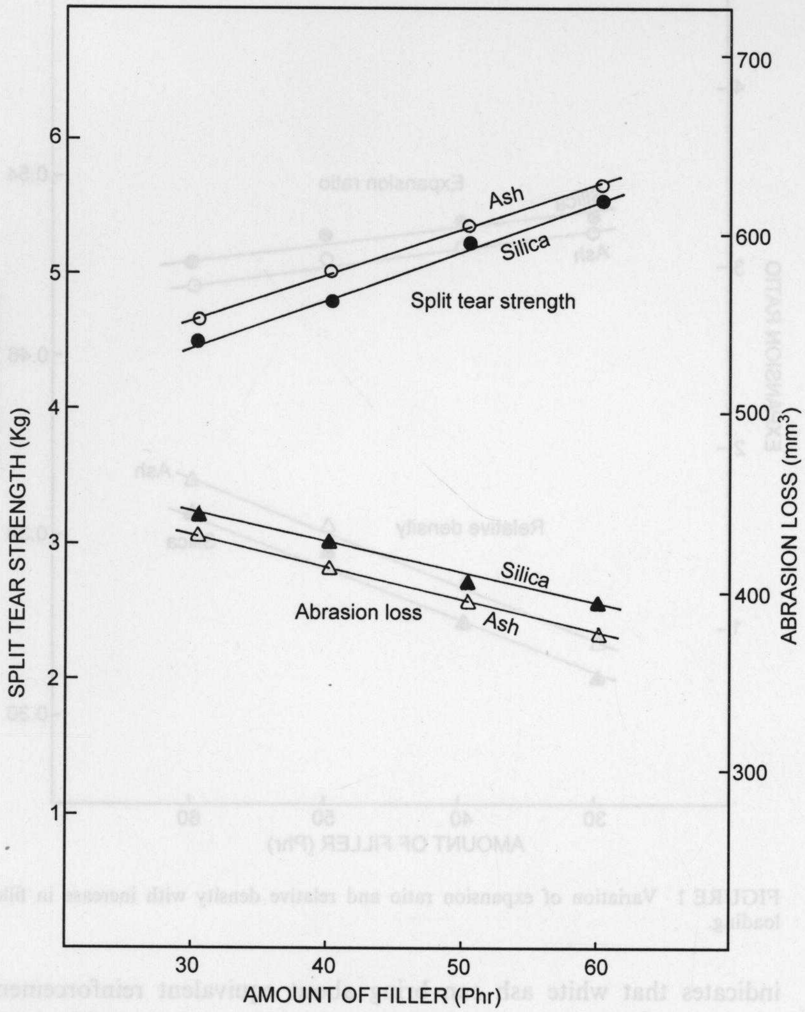

FIGURE 2 Variation of split tear strength and abrasion loss with increase in filler loading.

Hardness of the MC sheets increases with the addition of white ash. This increase in hardness is also due to the reinforcement of white ash. Heat shrinkage of the MC sheets is marginally reduced when white ash is used in place of silica. This is due to the lower expansion of the MC sheets.

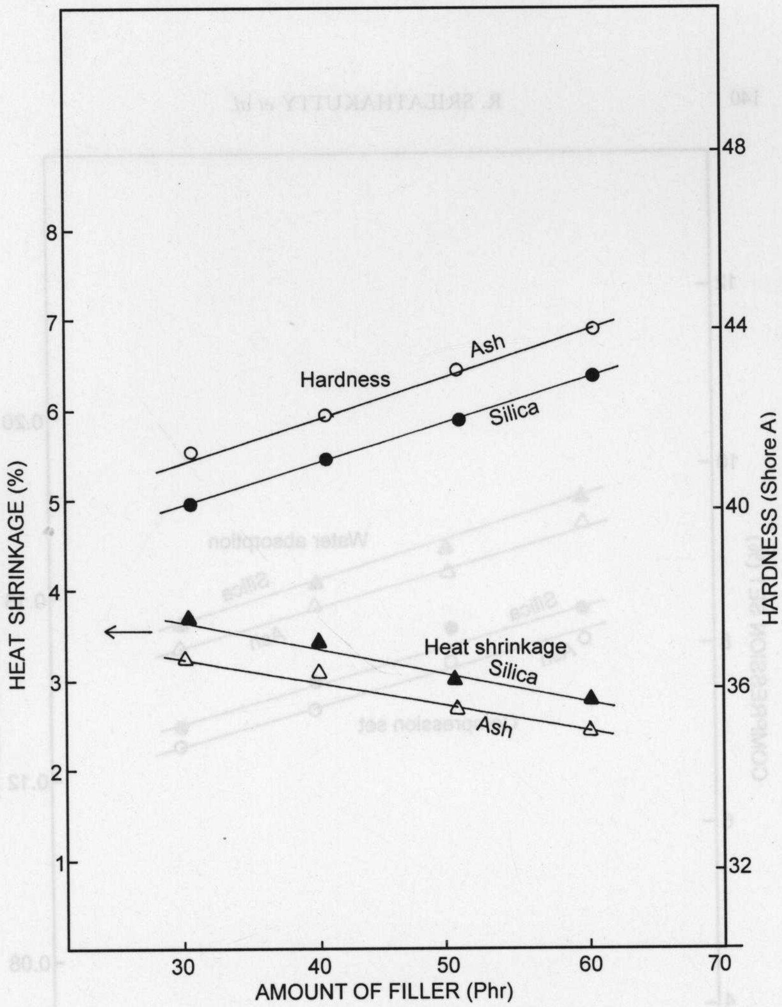

FIGURE 3 Variation of heat shrinkage and hardness with increase in filler loading.

Figure 4 shows the variation of compression set and water absorption in NBR/PVC based MC sheets. Compression set decreases when silica is replaced by white ash. This is also due to the stronger cell walls resulting from lower expansion of the MC sheets [5]. There is a reduction in water absorption when silica is replaced by white ash. This may be due to the lower percentage of open cells.

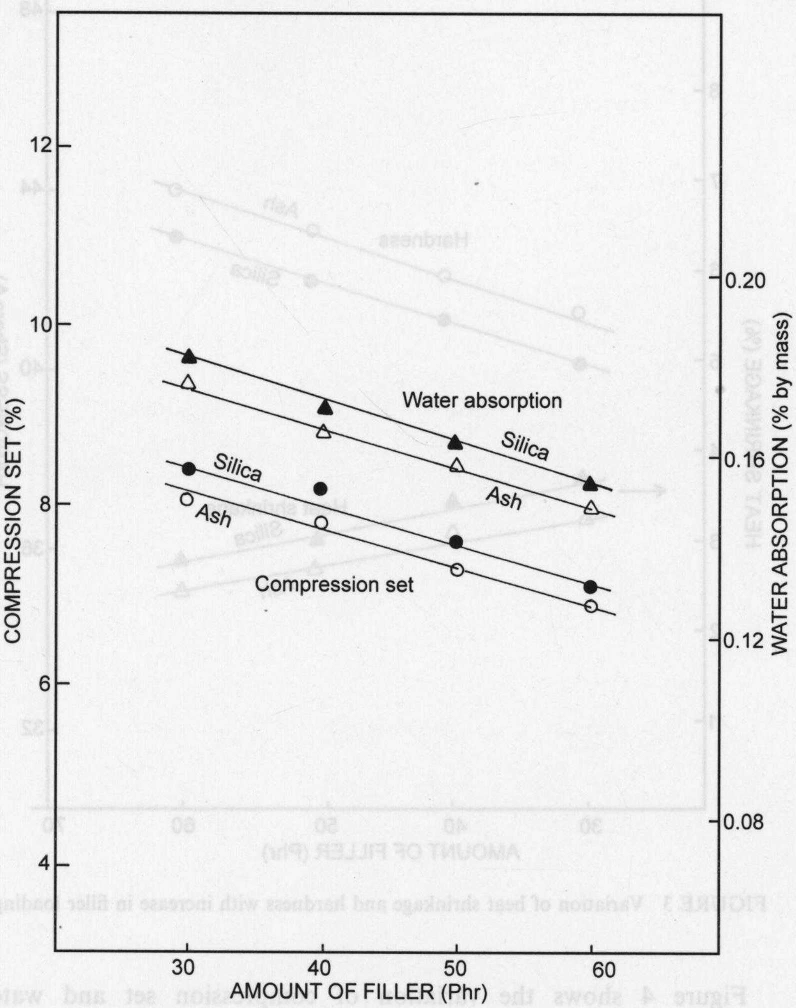

FIGURE 4 Variation of compression set and water absorption with increase in filler loading.

Cell Structure Studies

The optical photographs of razor cut surfaces of Microcellular rubber sheets are shown in the Figures 5 and 6.

Figure 5 is the photograph of the cell structure of microcellular soles containing 40 phr silica in NBR/PVC based MC soles. It can be seen

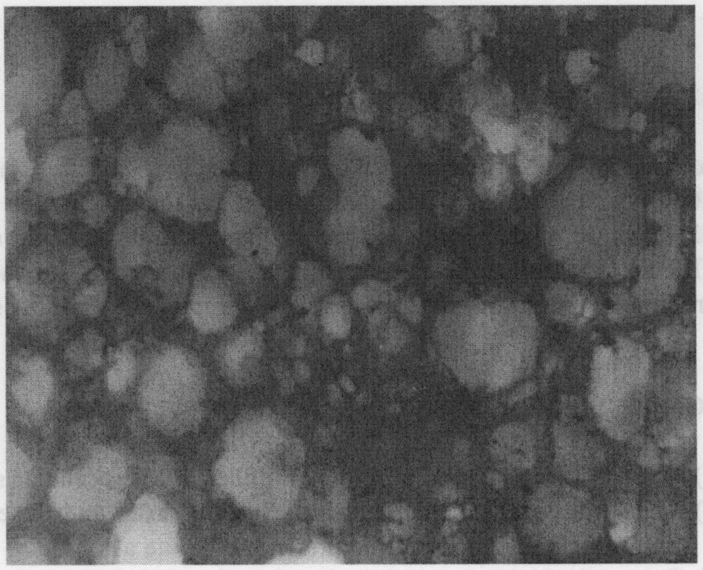

FIGURE 5 Optical photograph of the compound containing 40 Phr silica in NBR/PVC based MC sole compound.

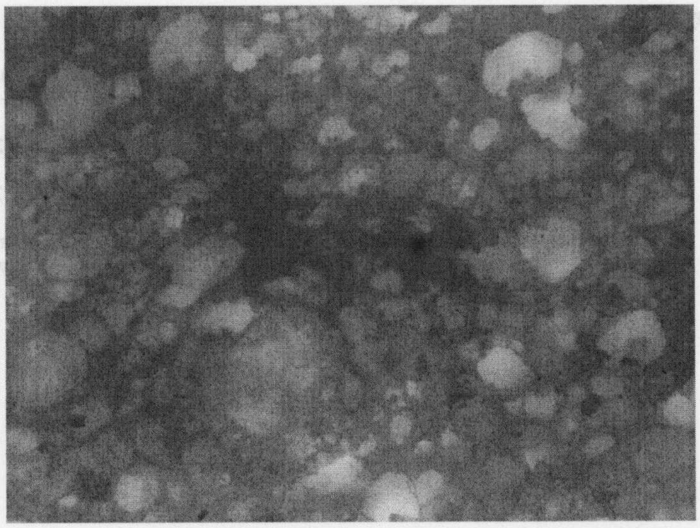

FIGURE 6 Optical photograph of the compound containing 40 Phr white ash in NBR/PVC based MC sole compound.

from the figure that uniform cell size is obtained when silica is used as filler in NBR/PVC based MC soles. Figure 6 is the photograph of the cell structure of microcellular sheets containing 40 phr white ash in NBR/PVC based MC sole. From the photographs it is clear that cell structure remains unchanged when silica is replaced by white ash.

CONCLUSION

(1) White ash can be used as a low cost filler in Microcellular soles.
(2) The curing of the compounds is marginally faster and the expansion marginally higher in the presence of white ash.
(3) Due to the higher expansion, the amount of blowing agent can be marginally reduced and this results in a product with improved mechanical properties.

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