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MICROCELLULAR CRUMBS IN PROFILE EXTRUSION

By

W. S. E. FERNANDO AND LI JIU-LONG*

SYNOPSIS

The use of sulphur cross linked unsaturated fats and oils, with natural rubber, referred to as factice, and superior processing rubbers, respectively, are recommended for minimising extrudate distortion in profile extrusion of natural and synthetic rubber compounds. It has been observed that microcellular scrap could also be used to achieve the same purpose after mechanical pulverisation.

INTRODUCTION

The retention of dimensional stability of the product and extrudate distortion are the two major problems encountered in profile extrusion. Factice, a sulphur cross linked unsaturated oil has been used over the years to partially alleviate these problems. During the 1960's the Rubber Research Institute of Malaysia introduced superior processing rubber (SP rubber) as a substitute for factice (Baker and Foden, 1960). SP rubbers contain a proportion of (20%) cross linked rubber as microscopic particles intimately dispersed in a matrix of unvulcanized rubber. A concentrated form of SP rubber called PA 80 containing only 20 parts of unvulcanized rubber was later introduced (Falloner, 1963). Factice and SP rubber when incorporated in NR or synthetic rubbers offer some or all of the following advantages (Fletcher and Banker, 1962 ; Hordella, 1969).

1. Reducing the nerve or elasticity in the uncured compound and hence:
   Speeds incorporation of fillers,
   Improves dispersion,
   Reduces extrusion die swell and linear shrinkage,
   Improves definition of extruded profile,
   Improves calendering gauge control and definition.

2. Minimising distortion of uncured compounds particulary in open cures in steam or air.

3. Lubricating the mix, giving :
   Lower power consumption,
   Cooler mixing and processing,
   Faster mixing.

* Rubber Department, Shandong Chemical Engineering Institute, Peoples Republic of China. Temporary worker at RRISL.

Furthermore, residual sulphur compounds in both factice and SP rubbers accelerate the vulcanization process. In addition to the above advantages the lower specific gravities (close to unity) help in reducing the compound costs.

Microcellular crumbs are obtained by mechanical disintegration of microcellular scrap. The latter being a sulphur cross linked material, it was envisaged that the crumbs would behave in a similar manner to SP rubber if the former can be dispersed effectively during compounding. This paper describes an attempt to assess the extrusion characteristics of NR compounds containing microcellular crumbs as a process aid.

EXPERIMENTAL

Materials

The microcellular crumbs and dark factice used in this investigation were provided by Bata Shoe Company (Sri Lanka) Limited, and Chemanex Limited, respectively.

Methods

The compounds (Tables 1 & 2) were mixed in the laboratory mixer with a fixed rotor speed of 30 rpm. The chamber and rotor were heated to 60°C before compounding. The compounding sequence was as follows :

Factice/Microcellular crumb only

Time (Mins)	Action
0	Add rubber
1	Add ZnO, antioxidant, stearic acid+ factice/crumb
3½	Dump

The accelerator and sulphur were added on the laboratory two roll mill, immediately after dumping.

With fillers

Time (Mins)	Action
0	Add rubber
1	Add ZnO, stearic acid, antioxidant, microcellular crumbs (+ HAF black) oil
2½	Add clay/whiting/vulcacil S
5	Dump

The accelerators and sulphur were added on the two roll mill immediately after dumping.

Extrusion characteristics were determined using a David Bridge Hot Feed Lab Extruder having an L/D ratio of 8.

The dimensions of the circular die are given below :

$$L = 3.45 \text{ cms}$$
$$D = 1.16 \text{ cms}$$

Table 1. *Compounding formulations without fillers*

Formulation	I	II	III
RSS 3	100.0	100.0	100.0
ZnO	4.0	4.0	4.0
Stearic acid	2.5	2.5	2.5
Flectol H	1.0	1.0	1.0
Paraffin wax	1.0	1.0	1.0
MBTS	0.8	0.8	0.8
DPG	0.2	0.2	0.2
Sulphur	2.5	2.5	2.5
Factice	—	25.0	—
Micro crumbs	—	—	25.0

Test equipment used

The following test equipment was used in this investigation :

Monsanto Model M 100 Rheometer
Hounsfield Tensometer
SPRI Shearing Disc Mooney Viscometer

Calculations

For die swell measurements, the extrudate diameter was assessed by calculating the volume of one centimeter of extrudate, using the following equation :

$$\text{Diameter D of extrudate} = 2r = 2\sqrt{\frac{M}{G\pi}} \quad \text{Where,}$$

M = Weight of one (1) cm of extrudate, g
G = Density of extrudate g/cc

$$\text{Die swell \%} = \frac{D - D^{\circ}}{D^{\circ}} \times 100$$

D° = Die diameter

RESULTS AND DISCUSSION

Microcellular crumbs are obtained by mechanical disintegration of scrap left over after punching soles from microcellular expanded soling materials. The cellular structure of material (micro – pores) assists in the grinding process. The crunbs so obtained are sulphur cross linked rubber containing filler (various types). The particle size distribution of the crumbs used in this investigation are given in Table 3.

Table 2. *Compounding formulation with fillers*

Stock Formulation

RSS 3	100.0
Zn0	4.0
Flectol H	1.0
Stearic acid	2.5
Wax	1.0
Dutrex R	2.0
MBTS	0.8
DPG	0.2
Sulphur	2.5

Formulation	IV	V	VI	VII	VIII	IX	X	XI	XII	XIII
Whiting	50.0	50.0	50.0	—	—	—	—	—	—	—
Clay	—	—	—	50.0	50.0	—	—	—	—	—
Vulcacil S	—	—	—	—	—	30.0	30.0	—	—	
Poly ethylene glycol	—	—	—	—	—	1.5	1.5	—	—	—
HAF Black	—	—	—	—	—	—	—	30.0	30.0	30.0
Factice	—	25.0	—	25.0	—	25.0	—	—	25.0	—
Micro crumbs	—	—	25.0	—	25.0	—	25.0	—	—	25.0

Table 3. *Particle size distribution of micro-crumbs*

Particle size		% Composition
Larger than 1700 μ	(10 mesh)	46
1700 – 710 μ	(10 – 22)	42
710 – 500 μ	(22 – 30)	5
500 – 355 μ	(30 – 44)	3
< 355 μ		4

The effective specific gravity of crumbs was determined indirectly using formulations I and III in Table I. The value obtained for the crumbs under investigation was 1.32, indicating a ratio of mineral filler to polymer (which is a mixture of NR and high styrene resin) around 1 : 1.

The cure and extrusion characteristics of the compound and the physical properties of vulcanizates containing 25 parts by weight of factice and microcellular crumbs (as represented by formulations II and III) are given in Tables 4, 5 and 6, respectively.

Table 4. *Cure characteristics of compounds* II *and* III

Cure characteristic	Compound II	Compound III
Mooney viscosity ML (1+4) @ 100° C	21.5	38.5
Mooney scorch time @ 120° C, min	14	22
Rheometer scorch time t_2, min	5	8
Minimum torque, torque units	8	10
Rheometer cure time t_{95}, min	23	28

Table 5. *Extrusion characteristics of compounds* II *and* III
(*Feed temp* 65°*C. Temp at die* 85°*C*)

Extrusion characteristic	Compound II	Compound III
Compound density (g/cc)	0.94	0.96
Extrusion rate cm/min		
20 RPM	26	40
40 RPM	46	68
80 RPM	108	132
Average wt/g/cm	2.81	2.27
Average diameter of extrudate (cm)	1.95	1.73
Average die swell %	68.1	49.1

Table 6. *Physical properties of vulcanisates*
(Press cure 30 mins @ 140° C)

Property	Compound II	Compound III
Hardness IRHD	37	48
Modulus @ 100 (M 100) kg/cm²	5.0	7.0
Modulus @ 300 (M 300) kg/cm²	14.0	34.0
Tensile strength kg/cm²	220.0	230.0
Elongation at break (%)	700.0	600.0
Tear strength, kg	119.5	156.7
Resilience Lupke	60.0	75.0
After air oven ageing for 3 days at 70°C		
Change in hardness IRHD	+5	1
Retention of TS (%)	96	94
Change in EB (%)	100	100
After air oven ageing for 7 days at 70°C		
Change in hardness IRHD	+7	3
Retention of TS (%)	68	62
Change in EB (%)	92	87

The presence of fillers in microcellular crumbs is reflected in the compound Mooney specific gravity and hardness values of the vulcanizates. The compound containing crumbs shows a fair degree of reinforcement and better tear resistance than factice. The physical properties of both vulcanizates meet the material property requirements of ISO 3861 and BS 5121 for the lining of a dirt and sand blasting hose.

It is generally recognised that extrudate swell is due to the elastic recovery of the materials. The strain elastic associated with extrusion, consists of two contributions :

1. The recovery of elastic strain stored at the entrance of the die, and
2. The recovery of elastic deformation related to the shear flow within the die.

It has also been demonstrated that for a given material at constant temperature and shear rate, post extrusion swelling depends upon the die characteristics (Leblanc, 1981). Therefore, it follows that if the die dimensions are kept constant at a given temperature and shear rate, the resulting post extrusion swelling can be generally attributed to material characteristics.

In this particular instance the difference in the swelling behaviour could be attributed to the difference in the interaction of factice and crumbs on the elastomer. It is therefore possible to conclude from the die swell measurements that microcellular crumbs used in this study decrease the elastic recovery of the rubber to a greater extent than dark factice at equal concentration.

Apart from die swell, it could be observed that the compound containing crumbs shows a faster rate of extrusion with no appreciable surface distortion, in gum compounds as well as in the presence of various types of fillers, Tables 5 and 7 – 10. The physical properties of vulcanizates obtained in the presence of fillers and microcrumbs will be dealt with in a separate publication.

Table 7. *Extrusion characteristics, in the presence of Filler-Whiting*

	Compound IV	Compound V (with factice)	Compound VI (with crumbs)
Compound density g/cc	1.0893	1.0687	1.1029
Extrusion rate at 80 RPM cm/min	140.2	162.4	186.2
Weight of extrudate gm	350.8	387.8	384.4
Diameter of extrudate cm	1.71	1.68	1.54
Die swell %	47.4	44.8	33.0

Table 8. *Extrusion characteristics in the presence of Filler–Clay*

	Compound VII (with factice)	Compound VIII (with crumbs)
Compound density	1.078	1.267
Extrusion rate at 80 RPM, cms/min	176.6	179.0
Weight of extrudate gms	365.8	350.8
Diameter of extrudate cm	1.56	1.40
Die swell%	34.7	20.7

Table 9. *Extrusion characteristics in the presence of Filler–Sillica (Vulcacil S)*

	Compound IX (with factice)	Compound X (with crumbs)
Compound density g/cc	1.0353	1.0631
Extrusion rate at 80 RPM cm/min	163.8	180.0
Weight of extrudate g	356.2	347.2
Diameter of extrudate cm	1.64	1.52
Die swell %	41.3	31.0

Table 10. *Extrusion characteristics in the presence of Filler–HAF Black*

	Compound XI	Compound XII (with factice)	Compound XIII (with crumbs)
Density of compound g/cc	1.035	1.056	1.096
Extrusion rate at 80 RPM cm/min	156.2	166.0	181.6
Weight of extrudate g.	331.2	340.6	345.6
Diameter of extrudate cm	1.64	1.57	1.48
Die swell %	41.3	35.5	28.1

Various types of surface irregularities of extrudates have been linked to elastic turbulence phenomena in the die entrance zone, brought about when shear exceeds a certain critical value (Leblanc, 1981). The extrusion output naturally increases with the shear rate. The compounds containing microcellular crumbs are seen to be capable of increasing the critical value of the shear rate thereby increasing the rate of extrusion without appreciable extrudate surface distortion (Plate I).

The Plate II shows the appearance of smooth walled and ribbed tube hoses extruded using formulation XIII. These suggest the feasibility of extruding tube hose with wall thickness around 2.0 mm in commercial scale, using microcellular crumbs to reduce extrudate distortion and improved dimensional stability.

CONCLUSION

Re-usable vulcanized crumbs could be easily obtained by mechanical size reduction from microcellular soling scrap. This breakdown requires less energy compared to other vulcanised material due to the presence of microscopic pores in the soling material.

It was observed that the incorporation of microcellular crumbs as a compounding ingredient increased the rate of extrusion, accompanied by lower die swell and linear shrinkage of natural rubber compounds, both in the presence and absence of fillers.

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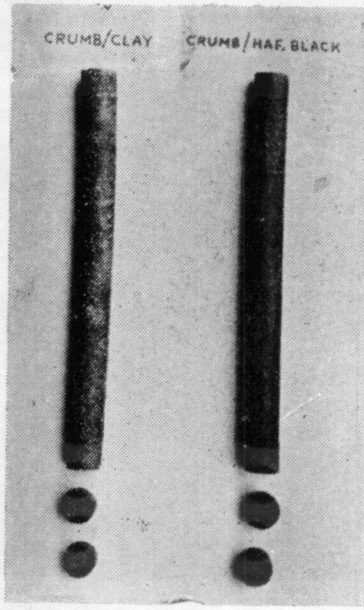

PLATE I. Extruded solid rods

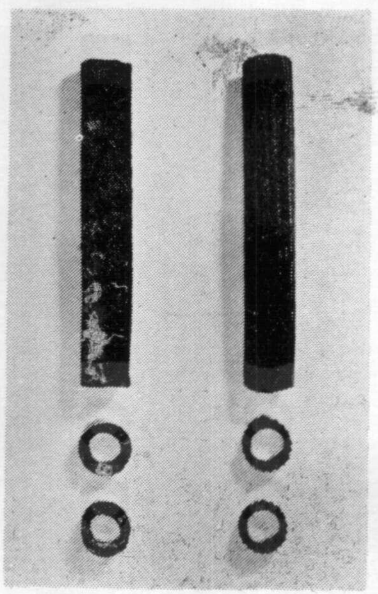

PLATE II. Extruded tube hoses