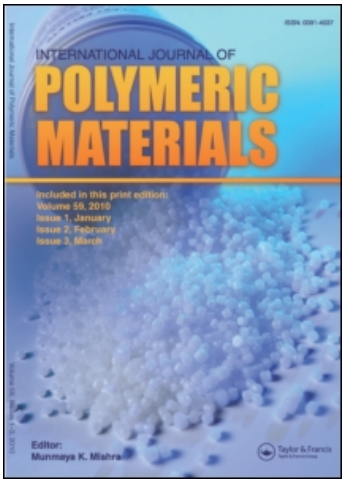

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Significance of Feeding Rate in the Extrusion of Filled and Gum IIR Vulcanizates

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Filled and gum compounds of Isobutylene-Isoprene rubber were extruded through a laboratory extruder at various feeding rates, different temperatures and revolutions per minute. The extruded compounds were vulcanized up to their optimum cure times and the mechanical properties of the vulcanizates were determined. The properties suggest that there is a particular feeding rate in the starved fed region, which results in maximum mechanical properties. The study shows that running the extruder at a slightly starved condition is an attractive means of improving the physical properties.

Keywords: Isobutylene-isoprene; extrusion; feeding rate; starved fed; mechanical properties

INTRODUCTION

Feeding of a polymer to a single screw extruder can be done by adopting any of the following methods.

(1) Starved feeding through the use of metering devices [1],
(2) Flood feeding through a hopper [2],
(3) Force feeding by using hopper compactor [3].

Among the above three approaches, starved feeding is commonly employed for improving the operating versatility of single screw

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extruders [4]. For example, the same screw can handle a wide range of materials and performance requirements often better utilizing horse power availability [5]. Further, starved feeding requires lower torque [6] and achieves higher stock temperature and increased variability in output rate [7]. In starved extrusion, the effective metering depth of the screw is reduced and it results in improvement in melting performance, due to the increase in rotational speed of the screw for the same screw rotation time [8]. Edwards *et al.* [9, 10] showed that the degree of mixing produced in the melting zone is more significant than that in metering zone, as in starved extrusion. So starved feeding is likely to result in reduced mechanical breakdown due to uniform mixing, better thermal homogeneity and preferential orientation of the molecules due to comparative decrease in melt temperature [11] which may give rise to improved mechanical properties. This study has been undertaken to determine the effect of feeding rate on the mechanical properties of filled and gum IIR vulcanizates.

EXPERIMENTAL

Materials

Isobutylene-Isoprene Rubber (IIR): Exxon 065, Mooney Viscosity ML (1 + 8) at 100 °C, 50; Supplied by Indian Petrochemicals Corporation Ltd.

Zinc Oxide, Stearic acid, Pilflex-13 [N-(1,3-Dimethyl butyl)-N-Phenyl-p-Phenylene diamine], Carbon black (HAF N330), Paraffinic oil, Benzothiazyl disulphide, Tetramethyl thiuram disuphide and sulphur used were commercial grade.

Preparation of Test Samples

Studies were done on a laboratory extruder attached to a Brabender plasticoder model PL 2000 with an L/D ratio of 10 and a compression ratio of 1 and provided with a feeding roll. The formulations of filled gum compounds of IIR selected for the study are shown in Table I.

Compounds were prepared on a laboratory two roll mill according to ASTM D 3184 and 3189 (1973). The compounds were sheeted out

TABLE I Formulations of the compounds

	IIR-Gum Compound	*IIR-Filled compound*
IIR	100	100
ZnO	4	4
Stearic Acid	2.0	2.0
Anti Oxidant (Pilflex-13)	1.0	1.0
HAF Black (N 330)		45.0
Paraffinic Oil		7.5
MBTS	0.5	0.5
TMTD	1.0	1.0
Sulfur	1.5	1.5

by passing though a 1 mm nip of the mixing mill. The sheets were cut into 10 mm strips for feeding into the extruder. The feeding rates were adjusted by placing different number of layers of the strips on the feeding roll and measured by the rate of output of the extrudate. The compounds were extruded at varying feeding rates mainly in the starved fed regions at 20, 40, 60 and 80 rpm and at different temperatures. The cure curves of the IIR compounds before and after extrusion were taken on a Goettfert Elastograph model 67.85 according to ASTM D 1646 (1981) at 170 °C. The extruded samples were vulcanized up to their optimum cure times (90% of the time required for attaining maximum torque) in an electrically heated laboratory hydraulic press. The moulded samples were then cooled by immersing in water and dumb-bell specimens were cut out of the sheets for tensile testing. The tensile properties of vulcanizates were measured using a Zwick universal testing machine of model 1445 at an extension rate 500 mm/min as per ASTM D 412 (1980). The swelling index of the gum and filled vulcanizates were measured by equilibrium swelling in toluene [12] according to the following equation,

$$\text{Swelling Index} = \frac{\text{Final Weight} - \text{Deswollen Weight}}{\text{Initial Weight}}. \quad (1)$$

The viscosity values of a few sets of samples of gum rubber vulcanizates were measured by using a Brookfield viscometer. The percent bound rubber content (filler gel) of a few sets of samples were determined by immersing the samples in 25 ml of toluene for seven days at

room temperature (solvent was renewed after three days). Then the sample was dried for one day in air at room temperature and then for 24 hours in an oven at 105°C. The percent bound rubber of the polymer (RB) was then calculated according to the following equation [13]

$$RB = \frac{W_{fg} - W[m_f/(m_f + m_p)] \times 100}{W[m_p/(m_f + m_p)]} \quad (2)$$

where W_{fg} is the weight of carbon black and gel,
m_f is the weight of the filler in the compound,
m_p is the weight of the polymer in the compound and
W is the weight of the specimen.

The TGA curves of starved and normally fed IIR compounds were taken on a thermo gravimetric analyser, model TGA-50. The tensile fracture surfaces of a few typical samples were examined using a scanning electron microscope to study the mode of failure.

RESULTS AND DISCUSSIONS

Figure 1 shows the variation of tensile strength with feeding rate for IIR gum and filled vulcanizates at different rpm at 80°C. The highest feeding rate for each rpm represents the feeding rate recommended by the manufacturer. It is found that irrespective of rpm, the tensile strength initially increases with feeding rate, reaches a maximum value and thereafter decreases. This shows that for a given shear rate and temperature, there is a particular feeding rate in the starved region which results in maximum tensile strength. This is possibly due to the improved uniformity in temperature of the compounds [14], since thick sections are not properly heated to uniform temperatures in the case of thermal insulators like rubber. Further, the lower shear breakdown and preferential orientation of the molecules may be the other reasons for the higher tensile strength at this feeding rate [15]. The preferential orientation effect is clearly seen from the higher difference between the tensile strength measured in the longitudinal (extrusion) and transverse directions in this case, (Figs. 1 and 2).

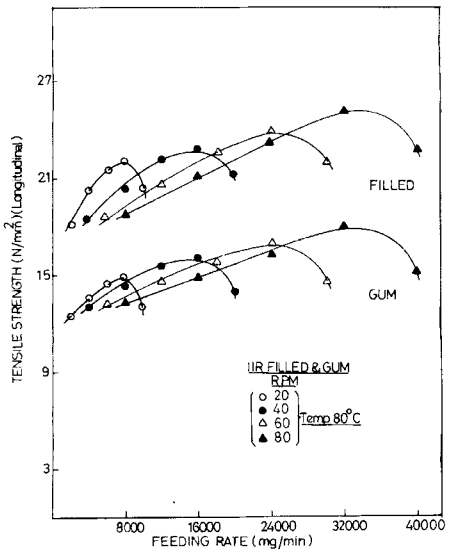

FIGURE 1 Effect of feeding rate on the tensile strength of gum and filled IIR vulcanizates at different rpm in longitudinal (extrusion) direction.

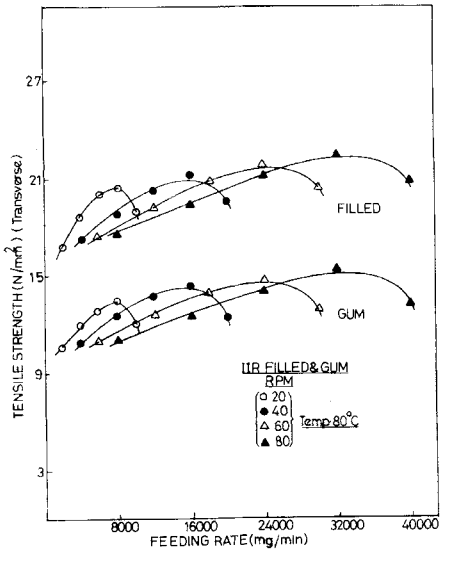

FIGURE 2 Effect of feeding rate on the tensile strength of gum and filled IIR vulcanizates at different rpm in transverse direction.

Figures 3 and 4 show the variation in elongation at break of IIR gum and filled vulcanizates with feeding rate in longitudinal (extrusion) and transverse directions at different rpm. As in the case of tensile strength, unimodel curves are obtained for each rpm. This further shows the efficiency of the starved extrusion in getting maximum physical properties.

Figures 5 and 6 show the variation in tensile strength of IIR gum and filled vulcanizates with feeding rate at different temperatures at a fixed rpm. The tensile strength improves with the temperature when the temperature is raised from 60 °C to 120 °C showing that the deterioration due to thermal degradation is not serious in this range. But the strength at 140 °C is less than at 120 °C in the case of gum and filled IIR vulcanizates showing the onset of degradation. At every temperature, the strength increases with feeding rate, reaches a maximum and decreases thereafter. As before, the maximum occurs just below the feeding rate suggested by the manufacturer in the range 60–120 °C and further down at 140 °C in the case of gum and filled IIR vulcanizates.

Figure 7 and 8 show the variation of elongation at break with feeding rate at different temperatures at a fixed rpm in the longitudinal

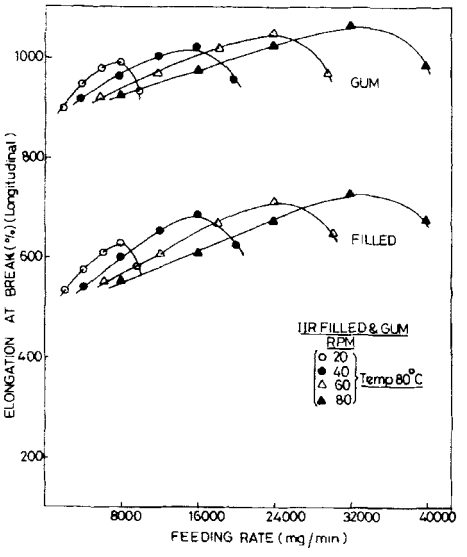

FIGURE 3 Effect of feeding rate on the elongation at break of gum and filled IIR vulcanizates at different rpm in longitudinal (extrusion) direction.

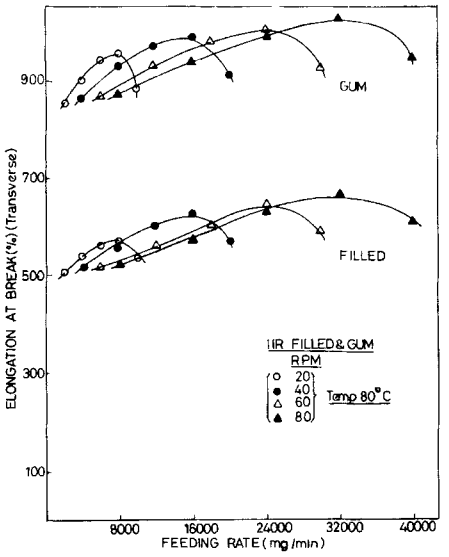

FIGURE 4 Effect of feeding rate on the elongation at break of gum and filled IIR vulcanizates at different rpm in transverse direction.

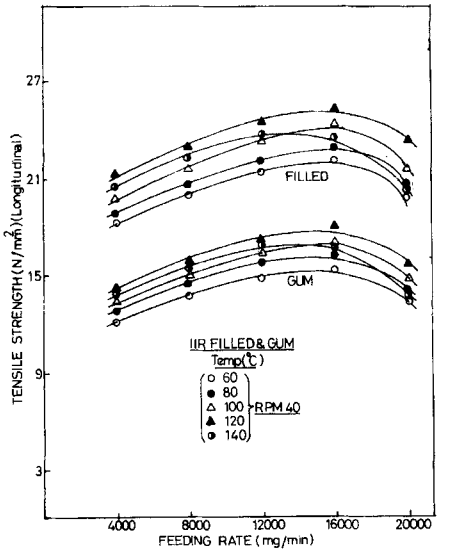

FIGURE 5 Effect of feeding rate on the tensile strength of gum and filled IIR vulcanizates at different temperatures in longitudinal (extrusion) direction.

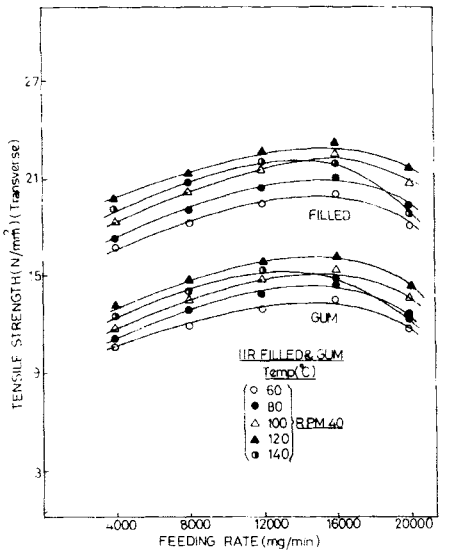

FIGURE 6 Effect of feeding rate on the tensile strength of gum and filled IIR vulcanizates at different temperatures in transverse direction.

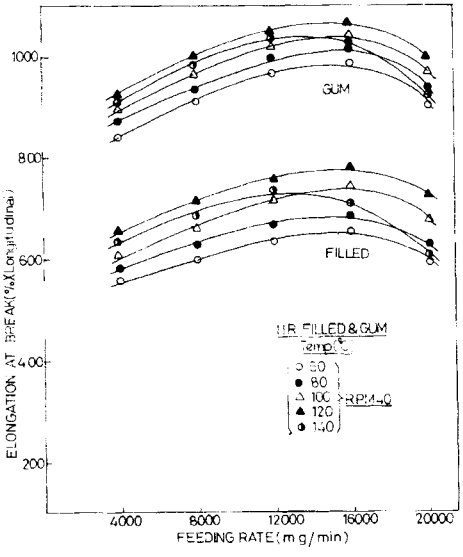

FIGURE 7 Effect of feeding rate on the elongation at break of gum and filled IIR vulcanizates at different temperatures in longitudinal (extrusion) direction.

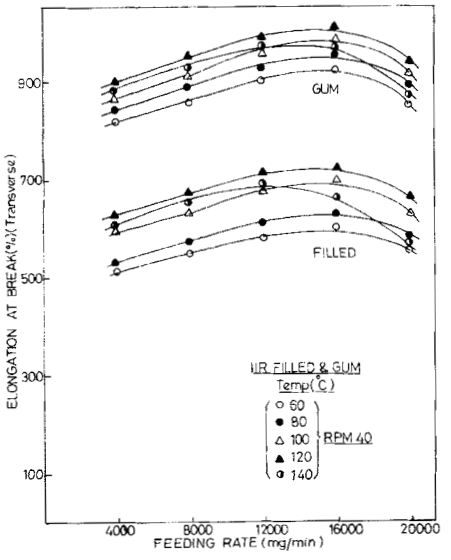

FIGURE 8 Effect of feeding rate on the elongation at break of gum and filled IIR vulcanizates at different temperatures in transverse direction.

and transverse directions. The behaviour is more or less similar to those of the variations of the tensile strength.

Table II shows the variation in swelling index and Brookfield viscosity values of gum IIR vulcanizates. It may be observed that the samples which give maximum physical properties show the least swelling index, i.e., maximum crosslink density obtained by the vulcanizates in the starved fed region. Moreover, starved extrusion results in higher viscosity values of the solutions due to lower mechanical breakdown.

Table III shows the variation of swelling index and percentage bound rubber content of the filled IIR vulcanizates. The least swelling index value, i.e., maximum crosslink density is observed for the extruded samples in the starved fed region. Further, the maximum in physical properties is obtained for the vulcanizates having the maximum bound rubber content in starved extrusion. This behaviour is expected since bound rubber has long been recognised as an important factor in the proper rubber-filler interaction and hence in rubber properties [16]. The TGA curves (Figs. 9 and 10) show that the change is endothermic and the starved sample has higher transition

TABLE II The variation of swelling Index and Brookfield viscosity values of gum IIR vulcanizates

	Feeding Rate in mg/min	*Swelling Index*	*Viscosity in Centipoise*
Unextruded IIR		2.16	1685
IIR extruded at rpm = 80	8000	1.88	1712
	16000	1.79	1736
Temperature = 80 °C	24000	1.65	1752
	32000	1.58	1788
	40000	1.84	1744
IIR extruded at rpm = 40	4000	2.27	1508
	8000	1.97	1524
Temperature = 120 °C	12000	1.89	1536
	16000	1.70	1572
	20000	2.01	1540

TABLE III The variation of swelling Index and percentage bound rubber content values of filled IIR vulcanizates

	Feeding Rate in mg/min	*Swelling Index*	*Bound Rubber Content %*
Unextruded IIR		2.00	27
IIR extruded at rpm = 80	8000	1.83	32
	16000	1.52	37
Temperature = 80 °C	24000	1.44	43
	32000	1.25	51
	40000	1.48	34
IIR extruded at rpm = 40	4000	1.62	36
	8000	1.50	39
Temperature = 120 °C	12000	1.36	46
	16000	1.20	57
	20000	1.43	41

temperature than the normally fed sample. This shows that the starved fed compound is more thermally stable than the normally fed sample. This behaviour probably results from the more uniform temperature and shear history to which the compound was subjected to in

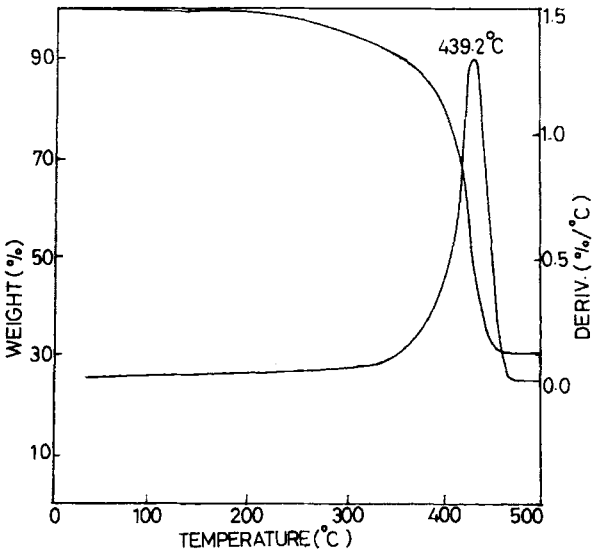

FIGURE 9 TGA curve of starved fed IIR sample.

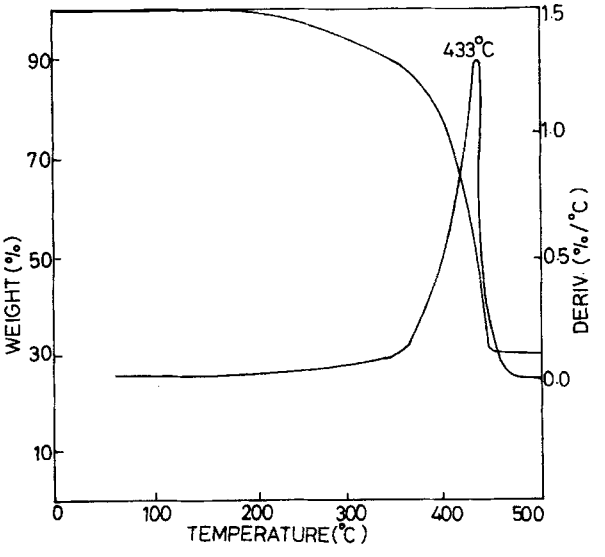

FIGURE 10 TGA curve of normally fed IIR sample.

starved extrusion than the normal extrusion. The tensile fracture surface of the starved fed vulcanizate is compared with those of the normal fed and unextruded samples in Figures 11a to c. The starved

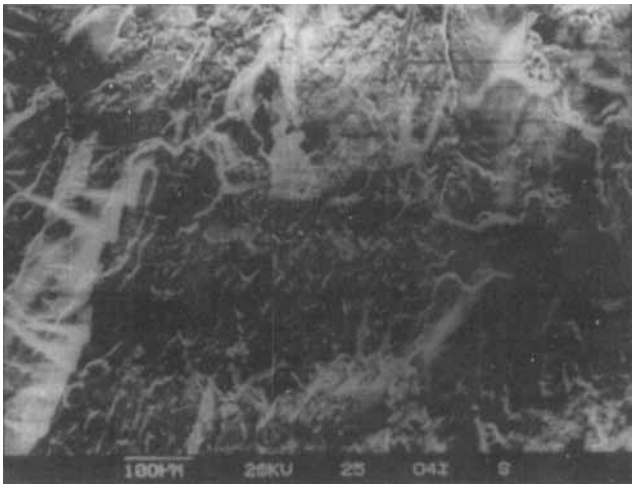

FIGURE 11(a) SEM photograph of IIR starved extruded sample.

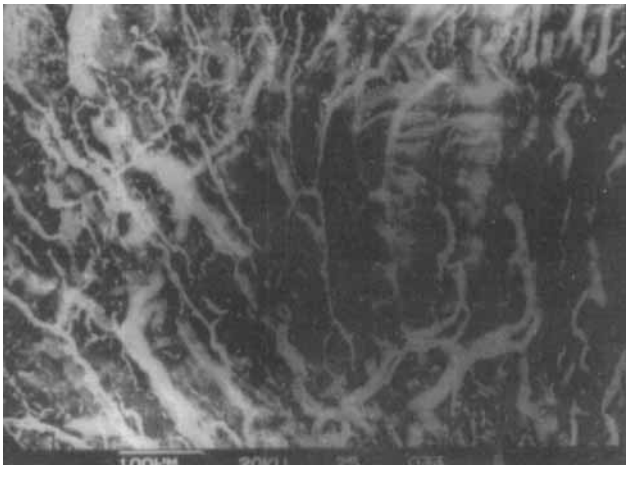

FIGURE 11(b) SEM photograph of IIR normally extruded sample.

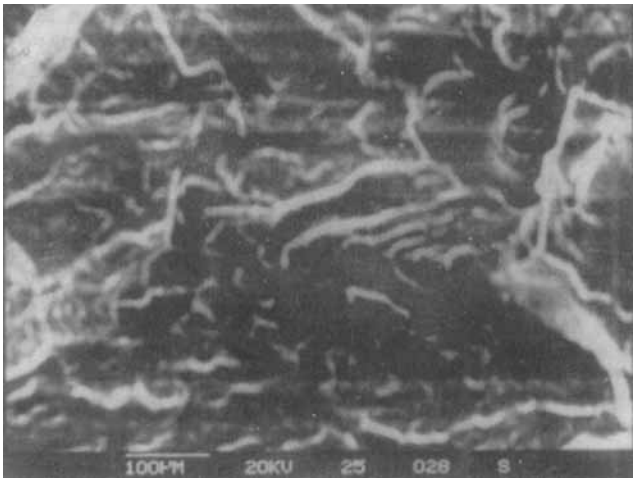

FIGURE 11(c) SEM photograph of IIR Unextruded sample.

fed sample is different from the other two showing its superiority possibly due to more uniform temperature distribution.

CONCLUSION

Starved feeding of IIR filled and gum compounds results in better heat transfer characteristics, proper preferential orientation, less mechanical breakdown and hence in a more uniform crosslink density and rubber-filler interaction. For a given screw, there is an optimum feeding rate in the starved fed region which results in maximum physical properties. So running an extruder at a slightly starved condition is an attractive means of improving the physical properties in addition to running the extruder at a lower torque.

References

[1] McKelvey, J. M. and Steingisten, S. (June 1978). *Plast. Eng.*, **7**, 51–54.
[2] Franszkoch, B. and Menges, G. (July 1978). *Plast. Eng.*, **10**, 51–54.
[3] Lovegrove, J. G. A. (December 1979). Plast. and Rubb. *Process*, **12**, 125–128.
[4] Maddock, B. H. (1959). SPE-15th *ANTEC*, **5**, 211.
[5] Russel. J. Nichols., George, A. and Kruder. (1974). *SPE Journal*, **20**, 462.

[6] Russel. J. Nichols., George, A. and Kruder. (1974). *SPE Journal*, **20**, 463.
[7] James. F. Stevenson. in Comprehensive Polymer Science "Speciality polymers and polymer processing" Sir. Geoffrey Allen., John, C. Bevington., and Sunder, C. Agarwal. (Eds.), Pergamon Press, Oxford, **7**, 152, (1991).
[8] Richardson, M. O. W. and Latif, L. H. (1991). *Progress in Rubb. and Plast. Tech.*, **7**, 152.
[9] Edwards, M. F., Gokbora, M. N. and Zayadine, K. Y. (1982). "Mixing Studies using a split barrel extruder", *Polymer Extrusion Conference*, London.
[10] Edwards, M. F. and Shales, R. W. (April 1985). "Mixing processes in single screw extruders", *I Chem. E. Symposium Series*, **94**, London.
[11] Richardson, M. O. W. and Latif, L. H. (1991). *Progress in Rubb. and Plast. Tech.*, **7**, 155.
[12] Ismail, H., Freakley, P. K., Bradley, R. H., Sutherland, I.and Sheng, E. (1995). *Plastics. Rubber and Compsites Processing and Applications*, **23**, 44–45.
[13] Siegfried Wolff., Meng Jiauwang.and Ewe-Hongtan. (1993). *Rubber Chem. Technol.*, **66**, 164.
[14] Joseph Francis, P. J., Joseph Rani and George, K. E. paper presented at Kerala Science Congress (January 1996) held at Cusat, Kochi, India.
[15] Joseph Francis, P. J., Joseph, Rani and George, K. E. paper presented at the International Conference on Macromolecules, (December 1994) held at V. S. S. C, Trivandrum, India.
[16] Siegfried Wolff., Meng Jiauwang and Ewe-Hongtan. (1993). *Rubber Chem. Technol.*, **66**, 163.