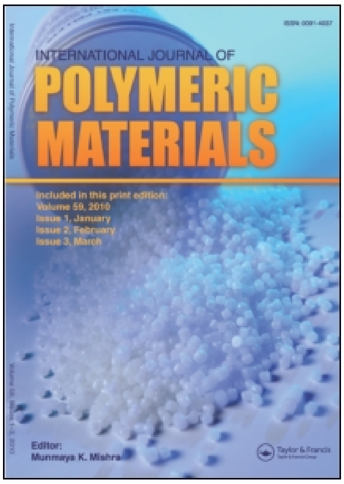

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K. T. Thomas[a]; N. M. Mathew[a]; R. Joseph[b]
[a] Rubber Research Institute of India, Kottayam, Kerala, India [b] Cochin University of Science and Technology, Kochi, Kerala, India

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Studies on the Processability of Different Forms of Natural Rubber Using Torque-rheometer

K.T. THOMAS[a], N.M. MATHEW[a,*] and R. JOSEPH[b]

[a] *Rubber Research Institute of India, Kottayam, Kerala : 686 009, India;*
[b] *Cochin University of Science and Technology, Kochi, Kerala : 682 022, India*

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Haake-Rheocord-90 instrument has been used to study the rheological behaviour of different forms of natural rubber. Since the design of the mixing head of the equipment is similar to that of and internal mixer, the behaviour of the rubber in actual processing can be estimated.

Keywords: Natural rubber; processability; rheological behaviour; torque-rheometer

INTRODUCTION

Rheological behaviour of raw and rubber compounds, play an important role in product manufacturing operations such as mixing, extrusion, calendering and moulding [1–2]. In order to predict the processability of an elastomer, test conditions should simulate the operating conditions as closely as possible. As the above operations involve very high shear rates, capillary rheometer or torque rheometer can provide more realistic conclusions on the rheological response of rubber during processing.

Earlier studies on processability of natural rubber have been based on parameters such as Mooney viscosity and plasticity [3–4]. A few

* Corresponding author.

studies on the rheology of raw rubber have also been reported [5 – 6]. The objectives of such studies were to acquire typical flow data for different grades of natural rubber and to compare the rheological behaviour of these forms.

Different processing conditions are adopted for sheet, crepe and technically specified rubber. The former two forms are dried at lower temperatures compared to technically specified rubber (TSR). The mechanical energy input in the production of TSR is also higher. This is reflected in the molecular parameters and plasticity of the different forms of NR [7].

In the present work the Haake-Rheocord-90 has been used to study the rheological behaviour of different forms of natural rubber. The instrument imparts a very complex shearing motion to the polymer and subsequently the data cannot be taken as that of fundamental rheological properties. However, the design of the mixing head of the equipment is similar to that of an internal mixer and hence the behaviour of the rubber in actual processing can be compared. The processability of different elastomers have been studied earlier using a Brabender Plasticorder [8 – 10].

EXPERIMENTAL

Different forms of natural rubber were collected from known sources. The raw rubber properties of the same are given in Table I. As Estate Brown Crepe (EBC) and Indian Standard Natural Rubber (ISNR 20) are processed from fresh and dry forms of field coagula both were included in the study. The samples were masticated in the Haake-Rheocord under the following conditions.

Specific volume	–	259 cc
Temperature	–	80°C
Revolution	–	60 min^{-1}
Time	–	15 min.

The different forms of NR were characterized by the rheogram. The nature of the torque and temperature curves and their changes with time are characteristic of the material. To analyse the changes

TABLE I Raw rubber properties

Form of rubber	*Wallace plasticity*	*PRI*	*Ash%*	*Volatile matter %*	*Dirt %*	*Nitrogen %*
RSS 4	46	81	0.63	0.92	0.03	0.56
EBC (FC)	53	64	0.57	1.25	0.20	0.48
EBC (DC)	54	56	0.72	1.61	0.60	0.40
ISNR 20(FC)	52	60	0.63	0.46	0.13	0.38
ISNR 20(DC)	41	57	0.71	0.37	0.11	0.45

occurring during mastication, the following parameters, defined earlier, were evaluated.

M_2 (Nm)	–	The value of the torque at the 2nd minute.
M_{15} (Nm)	–	The value of the torque at the 15th minute, *i.e.*, the final torque.
M_{2-15} (Nm)	–	The reduction in the value of the torque.
T_F (°C)	–	Final temperature
ΔT°C	–	Difference between the initial and final temperature
W_{2-15} (Nm – min)	–	The energy input from 2 to 15 min, which can be obtained directly from the difference in the corresponding TTQ values.

The samples were masticated under the specified conditions and the above parameters were measured. The masticated raw rubber samples were matured for 24 h and again subjected to a second cycle of mastication, under the same conditions. Figures 1 and 2 show typical curves obtained for EBC (DC) and RSS 4 samples respectively for successive mastication tests. Table II gives the values for the first cycle of mastication. The highest M_2 value is obtained for RSS 4 samples followed by EBC (FC) samples. During the process of mastication, the torque and temperature attains almost a steady state which is characterized by M_{15} and T_F. It can be seen that the final temperature is directly proportional to M_{15}. The largest reduction in torque *i.e.*, M_{2-15} was observed for EBC (FC) samples followed by ISNR 20(FC) samples. Increase in temperature *i.e.* (ΔT) is found to be proportional to the reduction in viscosity. However, in the case of RSS 4, ΔT is the highest with comparatively lower value for M_{2-15}. This could be

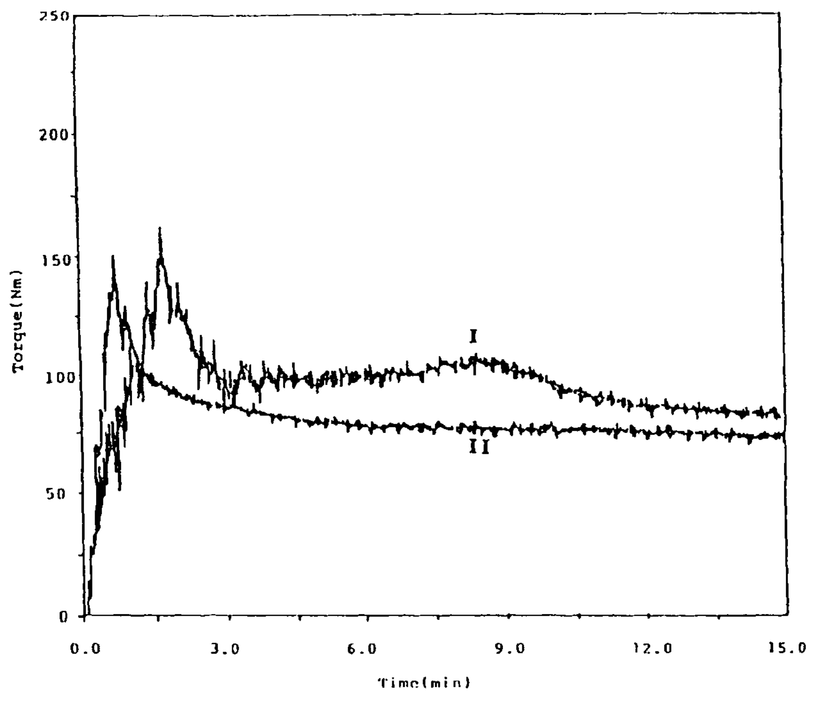

FIGURE 1 Typical Rheogram obtained for EBC (DC) sample during successive mastication.

attributed to the higher molecular weight of RSS 4 sample, as it undergoes least degradation during processing. Hence the viscoisty reduction in this case is less. In the case of field coagulum grades *viz.*, EBC (FC) and ISNR 20 (FC), M_{2-15} values are higher but ΔT is lower than that for RSS 4. ISNR 20 (DC) samples give the lowest value for M_{2-15}. W_{2-15}, *i.e.*, the work done in masticating the samples from the 2nd minute to the 15th minute, as measured by the TTQ, is higher for RSS 4, followed by EBC grades, ISNR 20 samples show lower values.

The values obtained for the defined parameters during the second mastication cycle are given in Table III.

The extent of variation of the values for the said parameters for both cycles can be taken as a measure of the degradation to the sample. A higher difference between the two successive cycles of mastication indicates more degradation of the rubber at the molecular

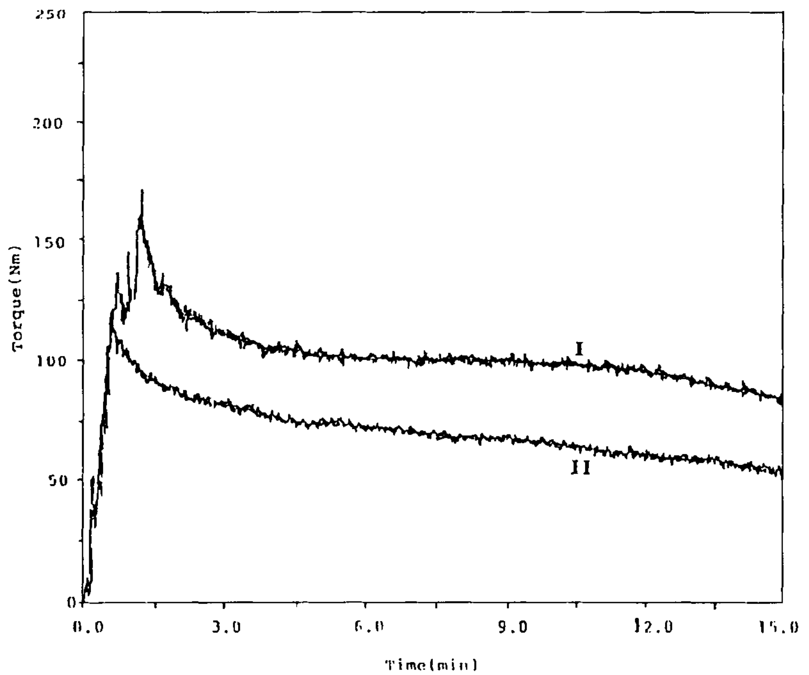

FIGURE 2 Typical Rheogram obtained for RSS4 during successive mastication.

TABLE II Characteristic values for 1st cycle of mastication

Form of rubber	M_2 (*Nm*)	M_{15} (*Nm*)	$T_F(°C)$	M_{2-15}	ΔP_0	$\Delta T(°C)$	W_{2-15} (*Nm–Min*)
RSS 4	128	84	158	44	16	78	1303
EBC (FC)	123	73	148	50	19	68	1240
EBC (DC)	125	79	148	46	15	65	1202
ISNR 20(FC)	123	74	146	49	17	66	1139
ISNR 20(DC)	100	68	142	32	12	62	1150

TABLE III Characteristic values for 2nd cycle of mastication

Form of rubber	M_2 (*Nm*)	M_{15} (*Nm*)	$T_F(°C)$	M_{2-15}	ΔP_0	$\Delta T(°C)$	W_{2-15} (*Nm–Min*)
RSS 4	77	55	131	22	10	51	859
EBC (FC)	94	74	141	20	12	58	1064
EBC (DC)	85	63	130	22	13	58	975
ISNR 20(FC)	87	57	130	30	14	50	949
ISNR 20(DC)	71	50	128	21	9	51	939

level. Bartha et al. [14] have studied the degradation behaviour of different rubbers on a Brabender Plasticorder in a similar way. They have reported that butyl rubber showed minimum difference between successive mastications, indicating less degradation.

Table IV gives the differences in the above parameters for the rubbers between the two successive mastication trials. It is seen that RSS 4 obviously, maintains difference for the above values indicating a higher level of degradation compared to the other forms. This is clearly seen from the differences in M_{15}, T_F, ΔT and W_{2-15} values. ΔP_0 i.e., the change in P_0 values of the same at the 2nd minute and 15th minute of mastication can be correlated with M_{2-15} values. The EBC (FC) samples show similar behaviour but less significant compared to RSS 4.

The least difference is shown by EBC (DC) samples, as the extent of degradation occurred to the material during the second mastication is minimum. This is because, the material had already degraded to a high level, so that mechanical shearing becomes less effective during mastication to cause further breakdown.

The torque (M) at a particular temperature can be taken as approximately equivalent to viscosity. Hence, Arrhenius equation can be applied and

$$\log M = Ae^{E/RT}$$

where M is the torque, E is the activation energy, R the universal gas constant and A a constant. A plot of $\log M$ vs $1/T$ can give straight lines, characteristic of each sample. The slope of the same can give a measure of the energy of activation, though the absolute values may be far from the values obtained from capillary rheometry.

The plot of $\log M$ vs $1/T$ made for EBC (DC) samples for the two mastication tests are given in Figure 3. The values of $(\Delta \log M/(\Delta 1/T))$

TABLE IV Difference in characteristic values during 1st and 2nd mastication cycles

Form of rubber	M_2 (Nm)	M_{15} (Nm)	T_F(°C)	M_{2-15} (Nm)	ΔP_0	ΔT(°C)	W_{2-15} (Nm-Min)
RSS 4	51	29	27	22	6	27	444
EBC (FC)	38	10	18	28	6	10	265
EBC (DC)	31	5	7	26	3	7	138
ISNR 20(FC)	36	17	16	19	3	16	190
ISNR 20(DC)	29	18	14	11	3	11	211

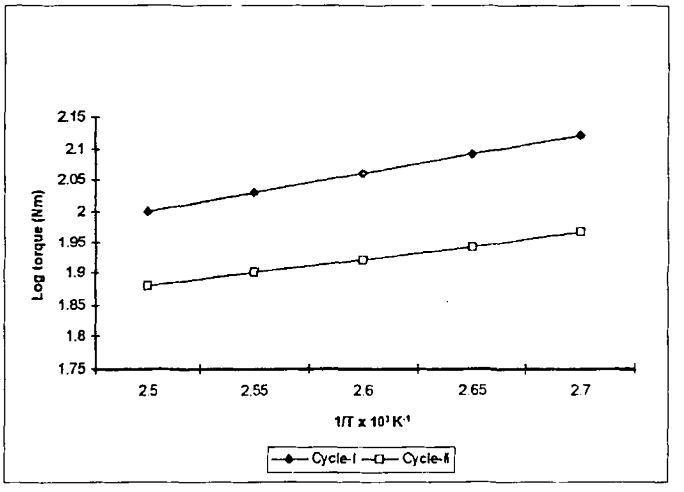

FIGURE 3 Variation of Log Torque with Temperature for EBC (DC) sample.

TABLE V ($\Delta \log M/(\Delta 1/T)$) values for different forms during successive mastication cycles $\times 10^3$

Form of rubber	*Ist cycle*	*IInd cycle*
RSS 4	0.86	0.54
EBC (FC)	0.74	0.51
EBC (DC)	0.48	0.41
ISNR 20(FC)	0.59	0.47
ISNR 20(DC)	0.53	0.49

are worked out for each sample for both the mastication tests and are given in Table V. It can be seen that the difference between the gradients is maximum for RSS 4 samples and minimum for EBC (DC) samples. EBC (FC) samples show higher levels of degradation though ISNR 20 samples (both grades) show almost similar values. The lower value for the gradient obtained for EBC (DC) agrees well with the lower values for energy of activation E obtained when the same were evaluated using the capillary rheometer.

CONCLUSIONS

1. The torque rheometer can give meaningful conclusions on the rheological behaviour of different forms of natural rubber.

2. The difference in the extent of breakdown parameters of the rubber, during successive mastication can be taken as a measure of the degradation.
3. A plot of log M *vs* $1/T$ can give straight lines characteristic of each sample. The slope of the same can give a measure of the energy of activation.
4. RSS 4 gives maximum difference between the slopes for Arrhenius plots and EBC (DC) the minimum, indicating higher extent of degradation for the former.
5. The lower values for the gradient of the Arrhenius plot, obtained for EBC (DC) agrees well with the lower values for energy of activation E, manifested by the same, using a capillary rheometer.

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