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A COMPARATIVE EVALUATION OF ISNR 20 WITH CONVENTIONAL FORMS OF NATURAL RUBBER

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A detailed study was undertaken to compare ISNR 20 with the conventional forms of natural rubber. Samples from different sources were collected periodically over a period of one year and the raw rubber, processing and vulcanizate properties were evaluated. It was found that the sheet grades show better raw rubber properties except volatile matter and initial Wallace plasticity than ISNR 20 and EBC IX. It has also been found to have comparable level of consistency in these properties with that of ISNR 20. The vulcanizate properties including ageing characterisitics have also been found to be better for sheets. Significant differences were not observed among ISNR 20 made out of raw materials collected from estate and non estate sector

Key words: Natural rubber, ISNR, RSS, EBC, Raw rubber, Processing properties, Ageing.

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INTRODUCTION

A major share of natural rubber is being consumed by the tyre sector. The consumption pattern varies for different forms of rubber. The conventional grades of natural rubber which find extensive use in the above sector are sheet (RSS 4 and 5) and crepe (EBC IX and 2X). During the last decade, many processing units have been established in India for the production of technically specified rubber (TSR) and the total production of the same during 1993-94 was 31240 metric tonnes.

In India TSR is generally marketed in conformity with the specifications laid down by the Bureau of Indian Standards under the general name Indian Standard Natural Rubber (ISNR). The major advantages of ISNR are claimed to be its improved consistency in properties and the availability of different grades to suit individual requirements of the end user. The market trends indicate that among the ISNR grades, ISNR 20 is the preferred grade for the tyre sector considering its cost and quality. Studies have been made comparing sheet and block grades of rubber processed from latex (Loo, Sin and Chin, 1978; Bristow and Sears, 1986). Kuriakose *et al* .(1975) studied the processability and vulcanizate properties of block rubbers with the conventional grades of natural rubber.

The present study has been evisaged to compare raw rubber, processing and technological properties of ISNR 20 with those of the conventional forms of natural rubber, with special reference to the level of consistency in these properties.

EXPERIMENTAL

Samples of RSS 4, RSS 5, EBC IX and ISNR 20 were collected from different regions in the traditional rubber growing areas four times with an interval of three

Table 1 **Sources of samples**

Type of rubber	Source
RSS 4, RSS 5	Palai, Venjaramood, Konni, Nilambur
EBC_1	Mundakayam, Nagercoil, Kulasekharam, Manimala
EBC_2	Different processing units in and around Kottayam.
$ISNR\ 20_1$	Punalur, Mundakayam, Nilambur.
$ISNR\ 20_2$	Palai, Thodupuzha, Poovarani and Kottayam.

1 - Fresh field coagulaum 2 - Dry field coagulum

months each. As ISNR 20 and EBC are processed from fresh and semi-dried forms of field coagulum, both the types were included in the study. The details of the same are given in Table 1.

The raw rubber properties specified for ISNR and acetone extractables were determined for the above samples according to the respective BIS procedures. The processing properties of the ACS 1 mix and the technological properties of the HAF (N330) black filled mixes (formulation given in Table 2), wer evaluated as per the relevant ASTM test procedures.

Table 2. **Formualtion of HAF filled mix**

Components	Part of weight
Natural rubber	100
Stearic acid	2
Zinc oxide	5
Naphthenic oil	5
HAF black (N330)	50
N-cyclohexyl-2 benzothiazole sulphenamide	0.7
Sulphur	2.5

The data were statistically analysed and the mean, coefficient of variation and the desirability measure were worked out. The mean value explains the average performance of the grade with regard to the properties and the coefficient of vairation estimates the consistency. A more consistent distribution has a lower coefficient of variation and vice-versa. In order to determine the desirability of different forms of NR with regard to properties, a desirability measure (DM) has been worked out. Desirability measure is the sum of the squares of deviations of the observed values from the desired value expressed as relative to the desired value.

$$\text{Coefficient of variation (CV)} = \frac{\sigma}{\bar{x}} \times 100$$

where

σ = standard deviation

$\bar{x}$ = mean

and

$$\text{Desirability measure (DM)} = \frac{\Sigma |y-y'|}{N}$$

Where Y = observed value

y' = desired value

N = No. of samples

Since the performance stability and nearness to the desired value are equally important, mean, coefficient of variation and DM should be observed simultaneously for a reliable comparative analysis.

RESULTS AND DISCUSSION

Raw rubber properties

Table 3 shows the statistically analysed data for raw ruber properties. As was expected, dirt content is minimum for sheet grades. Compared to estate brown crepe (EBC) from non-estate sector, absolute values are better for ISNR 20. The value of DM is higher for EBC_2 grades while it is more or less the same for EBC_1, and ISNR 20 grades. Superiority of sheet grades is very evident from the significantly lower absolute values.

Table 3. **Raw rubber properties**

Parameters	RSS 4	RSS 5	EBC_2	EBC_1	$ISNR\ 20_2$	$ISRN\ 20_1$
Dirt, %						
Mean	0.07	0.09	0.32	0.13	0.13	0.13
CV	57.40	47.83	52.28	54.48	42.59	76.08
DM	0.06	0.08	0.31	0.12	0.12	0.12
Volatile matter, %						
Mean	1.03	1.05	1.43	0.90	0.58	0.41
CV	31.41	31.43	34.43	39.36	31.26	41.99
DM	0.89	0.90	1.28	0.75	0.43	0.26
Ash, %						
Mean	0.44	0.45	0.79	0.53	0.63	0.60
CV	27.10	26.30	22.25	34.62	35.56	44.40
DM	0.29	0.30	0.64	0.38	0.48	0.45
Nitrogen, %						
Mean	0.48	0.47	0.40	0.37	0.46	0.45
CV	18.82	17.54	16.68	27.28	23.44	71.38
DM	0.30	0.29	0.22	0.19	0.28	0.27
Po						
Mean	44	46	51	53	40	44
CV	12.30	14.55	8.99	10.39	18.12	19.58
DM	4.9	7.3	11.1	23.6	4.9	8.4
PRI						
Mean	82	80	46	60	62	58
CV	12.43	9.48	34.24	25.8	11.89	19.99
DM	12.44	14.1	48.94	35.00	32.44	36.56
Acetone extract, %						
Mean	3.04	2.94	1.85	2.25	2.52	2.36
CV	13.50	12.21	18.62	22.21	10.93	17.50
DM	1.94	1.84	0.75	1.15	1.42	1.26

The more efficient drying methods of ISNR 20 grades impart lower volatile matter as is evident from DM values. But CV is comparable in all the forms. If field coagulum (FC) is processed fresh into crepe, lower volatile matter could be achieved, as is observed for EBC_1. Processing of fresh field coagulum leads to lower volatile matter in ISNR also. Wrapping of ISNR with polyethylene sheet might also contribute to the lower volatile matter. In the case of sheets, the higher volatile matter may be attributed to the lower temperature range in the smoke house and due to the removal of sheets from the smoke house before complete drying prompted by a visually dry appearance. Storage of bare sheets in humid places also causes moisture absorption.

The field coagulum grades of rubber, viz. EBC and ISNR 20, contain higher ash than sheet rubber. The processing of field coagulum in the fresh stage causes reduced ash content. EBC_1 is close to ISNR 20 grades with respect to ash. EBC_2 contains maximum ash. The trend is consistent as the CV value is found to be lower. However, DM is found to be the highest for EBC_2. Sheet rubbers have the lowest ash content. Even the CV is lower compared to ISNR 20. DM also is found to be markedly lower for sheet.

Absolute values indicate that the lowest nitrogen content is for the crepe samples and the highest for the sheet. There is reasonably good consistency in all the samples except for ISNR 20_1 DM value is found to be minimum for crepe.

During the formation of field coagulum through spontaneous coagulation and during its storage, considerable bacterial activity takes place leading to decomposition of proteins into soluble products, resulting in lower nitrogen content in crepe as well as ISNR grades.

Wallace plasticity (Po) is indicative of the processability of the rubber and the energy required for mastication. Though the crepe samples show consistency, as is evident from CV values, the mean values for Po are higher for the same. This may be attributed to the lower level of thermo-oxidative chain scission taking place in the rubber probably owing to the lower temperature of drying, compared to that of ISNR 20 and sheet. Mean values for ISNR 20 grades indicate that they are in the processable range of plasticity consistency with sheet grades are comparable for the above. DM values are minimum for RSS 4 and ISNR 20_2.

Plasticity retention index (PRI) is a measure of the resistance to oxidation of raw rubber. As the raw material used in the production of sheet rubber is fresh latex. PRI of the same is higher, as was expected. PRI of crepe rubber processed from dry field coagulum is comparatively lower indicating the inferior quality of the raw material used. There is not much difference between PRI of the two types of ISNR 20. The CV values are also comparable for the sheet and ISNR 20 grades whereas the same is higher for the EBC grades. DM is minimum for the sheet gra-des followed by ISNR 20 and EBC_2 grades.

As expected, acetone exractables are higher for the sheet grades, since the chances of degradation and leaching by water are less. The effect of storage of field coagulum on the acetone extractables is manifested by the lower value of the same for EBC_2 grades. The CV values are comparable for sheet and ISNR 20 grades.

Processing properties

Table 4 enlists scorch and cure characteristics of mixes prepared as per ACS 1 formulation from the above samples. It is seen that for the sheet grades, optimum cure time and scorch time are higher probably due to acid retention on the same. But evidently ISNR 20 grades show better consistency in both the properties, as is evident from the CV values. This is definitely an advantage of ISNR 20, as it is showing consistent cure characteristics. But DM is minimum for the sheet grades. The above difference in scorch and cure characteristics are not so predominant for the HAF-filled vulcanizates. This could be attributed to the neutralisation of residual acidity by HAF black. CV values are comparatively lower for sheet grades, but the differences are less significant, DM being maximum for EBC_2 grades.

Technological properties

The tensile properties of the HAF-filled compounds are given in Table 5. Modulus at 100 percent and 300 percent elongation and tensile strength are higher for sheet rubber. The CV values are comparable for all the three forms. However, ISNR 20 processed from fresh field coagulum evidently shows better consistency than the same from dry field coagulum, as is evident from the lower CV values. DM is minimum for sheet grades followed by EBC_1 and ISNR 20_1 grades. CV is generally comparable and DM is maximum for EBC_2. Elongation at break is comparable for all the three forms, DM and CV showing similar trends.

Table 4. **Processing properties**

Parameter	RSS 4	RSS 5	EBC_2	EBC_1	$ISNR\ 20_2$	$ISNR\ 20_1$
ACS 1 mix						
Scorch time at 120°C, seconds						
Mean	790	810	491	519	511	448
CV	39.91	32.30	42.12	37.84	20.30	21.47
DM	693.23	667.13	991.88	963.69	972.0	1035.44
Optimum cure time 150°C, seconds						
Mean	921	958	704	738	662	680
CV	23.26	19.11	18.35	18.77	6.17	12.61
DM	518.44	481.92	735.38	702.31	777.69	759.62
HAF-filled mix						
Scorch time at 120°C, seconds						
Mean	1027	1022	935	884	869	933
CV	24.27	14.45	9.34	17.10	13.10	16.08
DM	449.0	453.67	540.92	592.42	606.67	543.25
Optimum cure time at 150°C, seconds						
Mean	590	572	501	613	578	573
CV	9.78	9.46	5.76	14.37	6.80	7.07
DM	130.0	148.0	219.0	107.0	142.5	147.0

Table 5. **Physical properties**

Property	RSS 4	RSS 5	EBC_2	EBC_1	$ISNR\ 20_2$	$ISNR\ 20_1$
100% modulus, N/mm^2						
Mean	3.22	3.09	3.02	3.07	2.92	2.95
CV	12.89	6.0	10.83	7.13	15.32	5.95
DM	0.68	0.81	0.83	0.88	0.98	0.95
300% modulus, N/mm^2						
Mean	11.43	11.23	10.83	10.91	10.32	10.65
CV	9.09	3.23	6.40	3.19	15.44	6.35
DM	1.68	1.88	2.19	2.28	2.78	2.45
Tensile strength, N/mm^2						
Mean	24.43	24.58	23.77	22.80	23.04	23.53
CV	7.73	3.56	7.13	16.82	8.43	5.19
DM	2.57	2.42	4.2	3.23	3.96	3.48
Elongation at break, (%)						
Mean	579	596	607	604	619	602
CV	9.66	8.95	6.80	6.01	12.01	8.10
DM	118.58	120.5	111.83	109.33	107.88	114.33
Retention after ageing at 70°C, 14 days, %						
Mean	59.0	57.30	59.70	63.90	62.30	52.50
CV	9.74	15.03	21.0	17.08	19.6	20.9
DM	23.0	24.7	18.10	22.3	22.5	29.5
Retention after ageing at 100°C, 4 days, %						
Mean	27.18	24.82	22.64	21.0	20.73	17.09
CV	46.8	32.6	37.6	29.1	20.8	26.9
DM	25.82	28.18	32.0	30.36	32.27	35.91

Ageing behaviour

Ageing of the vulcanizates at 100°C shows better retention in strength properties for the sheet grades, but bettern consistency is observed for the ISNR 20 grades. The CV values are marginally lower for the above. DM is minimum for the sheet grades. At 70°C, the differences in ageing properties are observed to be less significant. DM is minimum for the EBC_2 grades. The concentration of naturally occurring antioxidants is likely to be more in sheet rubber as it has been prepared by coagulation of latex under controlled conditions. The creosotic deposits formed due to smoking enhances the same. Concentration of natural antioxidants is lower in the field coagulum grades as a result of bacterial coagulation. Moreover field coagulum undergoes degradation to varying levels during storage. This explains the poorer ageing characteristics of EBC and ISNR grades compared to sheet.

Table 6 summarises the vulcanizate properties of the above compounds. Sheet grades show better wear, set and crack initiation characteristics. This could be explained on the basis of the higher molecular chain lengths in sheet as these are subjected to minimum mechanical work during processing. The lower dirt content contributes to the better crack initiation characteristics compared to scrap grades. CV values are almost comparable for all the

Table 6. **Technological properties**

Property	RSS 4	RSS 5	EBC_2	EBC_1	ISNR 20_2	ISNR 20_1
DIN abrasion loss, mm^3						
Mean	84.73	83.44	100.22	98.54	95.49	93.70
CV	15.95	14.55	12.13	16.03	11.23	11.05
DM	20.38	19.09	35.87	34.19	31.14	29.43
Compression set at 70°C, %						
Mean	28.87	28.82	36.06	33.81	34.04	34.43
CV	14.63	11.59	12.86	7.96	15.98	16.83
DM	4.94	4.89	12.13	9.88	10.11	10.50
Heat build up at 50°C, Δ T, °C						
Mean	28.03	26.97	27.39	27.27	25.67	26.45
CV	10.35	9.58	11.04	11.36	16.14	6.14
DM	11.33	10.27	10.69	10.57	8.91	9.75
Resilience, %						
Mean	55.04	54.60	55.35	54.51	55.39	54.83
CV	4.50	5.14	5.90	5.46	5.38	1.72
DM	6.72	7.13	6.38	7.22	6.34	6.90
Crack initiation, K.cycles						
Mean	14.54	13.25	11.44	10.82	10.47	10.90
CV	32.90	24.99	23.32	47.37	32.62	41.33
DM	9.51	9.73	11.54	13.23	13.58	13.15
Crack failure, K.cycles						
Mean	65.7	55.5	46.6	48.9	56.5	51.7
CV	25.0	29.9	23.5	45.5	23.4	28.1
DM	20.02	30.16	39.07	31.68	29.21	33.97

grades with respect to abrasion and compression set properties. The absolute values of the above indicate the superiority of sheet grades over the field coagulum grades. DM is minimum for sheet grades. ISNR 20_1 and EBC_1 grades show higher values for CV with respect to flex cracking characteristics.

Heat build-up values are marginally lower for ISNR 20 grades and this could be attributed to the fact that the 100 percent and 300 percent moduli values are lower for the above. DM is also significantly lower for the same. However, the CV values are distributed over a narrow range.

CONCLUSIONS

Sheet rubber grades show better raw rubber properties than crepe and ISNR 20 except for valatile matter and plasticity. Sheet grades are observed to have almost comparable level of consistency in raw rubber properties compared to that of ISNR 20. ISNR 20 grades show better consistency in processing properties such as scorch and cure characteristics. Vulcanizate properties such as tensile strength and elongation at break are better for sheet grades compared to all other grades. Significant differences were not observed among ISNR 20 processed from dry or fresh coagula. EBC from fresh field coagulum is superior to EBC from dry field coagulum in raw rubber and vulcanizate properties. All the raw rubber properties are superior for ISNR 20 from dry field coagulum compared to EBC from dry field coagulum. The technological properties are also better for ISNR 20 from dry field coagulum. Volativle matter and Po are higher for EBC from fresh field coagulum compared to ISNR 20 from the same type of quaterial. Scorch and cure times are slightly higher for the former but better consistency is shown for the latter. The technological properties are comparable for both the grades. Although ISNR 20 was expected to be more consistent, at least with respect to raw rubber properties, the level of raw material blending used for making these grades do not bring about this.

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