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Investigation of correlation between rheological properties of rubber compounds based on natural rubber/styrene-butadiene rubber with their thermal behaviors

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Abstract

Background: Rubber compounds are widely used in many applications because of the properties they exhibit. The physical and mechanical properties of these blends are sensitive to small variations in the amount of the individual polymers used. Thermogravimetry is currently gaining wide acceptance as a method for compositional analysis of vulcanizates. Knowledge of the relationship among thermal behavior of rubber compounds with their rheological properties is important for the assessment of the optimum process conditions to produce materials that have required properties. The correlation of rheological properties of rubber compounds based on natural rubber/ styrene-butadiene rubber with their thermal behavior has been assessed using thermogravimetry analysis. Thermogravimetric method permits the analysis to be completed in a short time and requires only a small sample.

Results: Thermogravimetry derivative profile (DTG) of the uncured blends and their rheological properties were investigated. Differential derivative curves of uncured vulcanizate showed that the degradation of styrene-butadiene rubber takes place at a higher temperature than that of natural rubber. According to DTG curves, two useful factors were demonstrated. These factors were the peak height ratio of natural rubber/styrene-butadiene rubber and a new factor called 'normalized factor $\left(\frac{h_{\mathrm{NR}x}/h_{\mathrm{NR}_{100}}}{h_{\mathrm{SBR}_{100-x}}/h_{\mathrm{SBR}_{100}}} \right)$,' which could be correlated with the rheological properties of the blends.

Conclusions: The result showed that the rheological nature of samples had acceptable correlation with the factors obtained by thermal analysis method. In other words, in this work a simple and reproducible experimental method was developed to efficiently predict the rheological properties of rubber blends.

Keywords: Natural rubber, Styrene-butadiene rubber, Thermal properties, Rheological properties

Background

Thermogravimetry (TGA) and derivative thermogravimetry (DTG) methods have emerged as powerful thermo-analytical techniques to monitor physical and chemical changes in both natural and synthetic materials. TG-DTG analysis allows the analysis to be completed in a short time with small amount of sample [1-3].

A standard tire formulation for trucks as well as cars is a physical blend of natural rubber/styrene-butadiene rubber (NR/SBR) or natural rubber/butadiene rubber (NR/BR) blends. Natural rubber and styrene/butadiene rubber have been blended for a long time for many purposes such as lowering the compound cost [4,5]. A lot of studies have demonstrated the physico-mechanical properties of such blends [6,7].

The rheological and physico-mechanical properties of NR, BR, and SBR blends are sensitive to small variations in the amounts of individual polymers used. Thermogravimetric analysis has been employed extensively to identify the elastomeric compounds especially NR/BR, NR/SBR, and NR/SBR/BR blends. Specifically, differential thermal gravimetric analysis (DTG) has a considerable value for estimating the basic composition of vulcanizates [8-15].

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Table 1 Formulation of tire tread based on NR/SBR blend

Components		Value (phr)	Company
NR[a]	(STR 20/BN)	0-100	Thaihua, Bangkok, Thailand
SBR[b]	(Cis-1220)	100-0	Arak Petrochemical, Tehran, Iran
Carbon black	(N-330)	50	Pars Tire, Tehran, Iran
IPPD[c]	(Pilfex 13)	1.5	Nocil Ltd., Mumbai, India
Rio wax[d]	(Anti lux 65X)	2	Rhein Chemie, Mannheim, Germany
TMQ[e]	(Antioxidant RD)	1	Nanjing, Jiangsu, China
Stearic acid	(PLMAC 1600)	4	Acidchem., Penang, Malaysia, Malaysia
ZnO	-	4	Shekohie, Qom, Iran
S	-	1.1	Tesduck, Tehran, Iran
OBTS[f]	(Vulcacit/MOZ)	0.8	LG/Lanxess, Zwijndrecht, Belgium

[a]Natural rubber; [b]styrene-butadiene rubber; [c]N-isopropyl-N'-phenyl-paraphenylenediamine; [d]blend of selected paraffins and microwaxes; [e]2,2,4-trimethy-1, 2-dihydroquinoline;[f]N-oxydiethylene-2-benzothiazole sulfonamide. phr, parts per hundred rubber.

The DTG curves of samples are used as 'fingerprints' in the identification of many single elastomer or blends, finding that the peak height of the DTG curves is dependent on the amount of each elastomer in the blends [16].

Taghvaei et al. analyzed the correlation of DTG peak height ratio with rheological and physico-mechanical properties of vulcanizate NR-BR blend and weight percent of BR, using thermogravimetric analysis [17]. We have recently investigated the correlation of the factor obtained by TG-DTG curve (h_{NR}/h_{BR}) and aging time of selected NR/BR blend (60/40). The results have shown that the relationship between h_{NR}/h_{BR} of blends and aging time has been fitted to a polynomial equation type, with acceptable regration [18].

Understanding and predicting of rheological and physico-mechanical properties of rubber compounds are really a great challenge. In this work, the samples of tire treads formulation were made of NR-SBR blends of known composition. The relationship between peak height ratio with their rheological properties and weight percent SBR of vulcanized NR-SBR blends were studied using thermogravimetric analysis. In addition,

the correlation of normalized factor with their rheological properties and weight percent of samples were studied using thermogravimetric analysis as well.

The most important advantages of these techniques are its easiness in use and its availability over a wide range of experimental conditions when compared with other rubber testing methods.

Methods
Materials
The material specifications and the recipe that is used for preparing the vulcanizate tire tread samples are given in Table 1. The major end use of this formulation is in tire for passenger trucks and cars.

Apparatus
The compounds were prepared by a two-roll mill (6 × 18 in.; Wellshayang, Tainan, Taiwan). The mixing roller to speed ratio was 1.2:1.0 and the roller distance was 10 mm, which was gradually shortened to 5 mm. The molding conditions of rubber blends were determined from data

Table 2 The rheological properties of NR/SBR blends

SBR (%)	TS$_2$ (s)	TC$_{90}$ (s)	MH (kg/cm)	ML (kg/cm)	CRI (s^{-1})
0	205	422	5.51	1.10	17.824
20	246	482	5.65	0.96	18.657
30	272	555	6.07	1.24	14.717
40	268	684	6.34	1.24	13.236
50	307	733	6.48	1.51	12.917
60	374	754	5.79	1.37	12.293
70	320	858	6.75	1.79	13.097
80	427	787	5.93	1.51	12.547
90	426	906	6.62	1.79	11.514
100	491	869	6.20	1.79	13.712
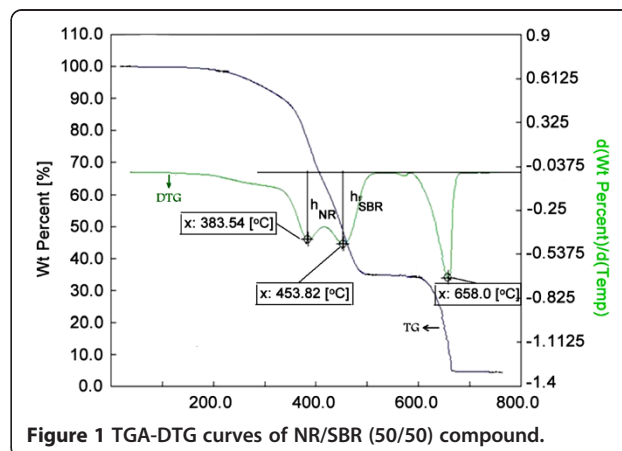
Figure 1 TGA-DTG curves of NR/SBR (50/50) compound.

Table 3 The factors obtained from the TG-DTG curves

SBR (%)	h_{NR}/h_{SBR}	Normalized factor $\left(\frac{h_{NRx}/h_{NR_{100}}}{h_{SBR_{100-x}}/h_{SBR_{100}}}\right)$
20	1.4	1.5
30	1.2	1.1
40	0.9	0.9
50	0.9	0.9
60	0.6	0.6
70	0.5	0.4
80	0.5	0.5
90	0.3	0.1

Figure 3 Plot of normalized factor vs. percentage of SBR.

obtained by a MDR 900, Hiwa Company (Shandong, China) at 160°C for 20 min. Thermogravimetric analysis was carried out using PL-TGA thermal analyzer according to ASTM D 6370-99.

Results and discussion

Rheological measurement

The rheological properties, such as the start of vulcanization scorch time (TS_2), optimum curing time (TC_{90}), cure rate index (CRI), minimum torque (ML), and maximum torque (MH) for different samples are shown in Table 2. Some of these properties were considered to correlate to the factors obtained by the thermal behavior.

Thermogravimetric analysis

Figure 1 shows TG-DTG curves for a blend with 50/50 ratio of NR/SBR. As shown in Figure 1, an initial weight loss between 100 and 200°C is due to the volatilization of oil and any other low boiling point components. The next weight loss is due to the decomposition of NR at 383°C and SBR at 453°C. Obviously, when the environment is switched from nitrogen to air, oxygen reacts with carbon black and leads to the last weight loss. It was found that the degradation of NR takes place in a

single step. Degradation of NR led to isoprene and dipentene and that of SBR led to a large number of products, like 4-vinyl cyclohexene, styrene, and methylbenzene. In the case of SBR, the degradation takes place in two stages. It was found as the SBR content increases, the DTG peak shifts to a higher temperature, indicating that the thermal stability of the samples was increased. As a result, it is possible to distinguish NR, which decomposes mainly at lower temperatures, from SBR. However, NR has a little weight loss around SBR decomposition temperature [16]. It is well known that the DTG curves are generally more sensitive to slight differences in weight loss than that of the parent curve; therefore, they can be used to correlate the thermal behavior of blends to their rheological properties. According to the DTG curves, two useful factors were demonstrated: peak height ratio of NR/SBR (h_{NR}/h_{SBR}) and a new factor called 'normalized factor $\left(\frac{h_{NRx}/h_{NR_{100}}}{h_{SBR_{10-x}}/h_{SBR_{100}}}\right)$' (Table 3).

As it has been observed from Figures 2 and 3, there is a linear correlation between weight percent of SBR to these two factors (Equation 1). On the other hand, there is satisfactory relation between the weight percentage of SBR and rheological properties of blends (Figures 4, 5, 6 and
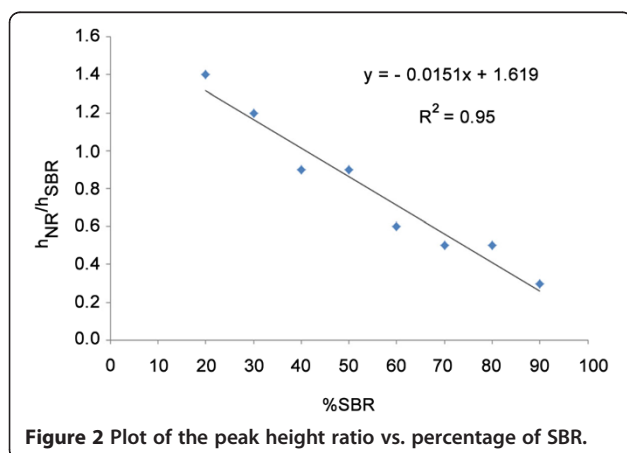
Figure 2 Plot of the peak height ratio vs. percentage of SBR.
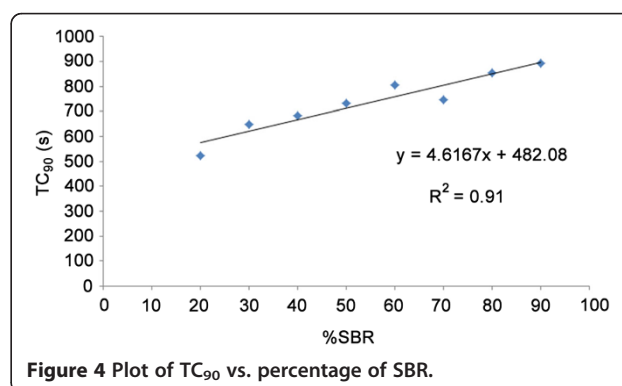
Figure 4 Plot of TC_{90} vs. percentage of SBR.

Figure 5 Plot of TS_2 vs. percentage of SBR.

Figure 7 Plot of TC_{90} vs. h_{NR}/h_{SBR}.
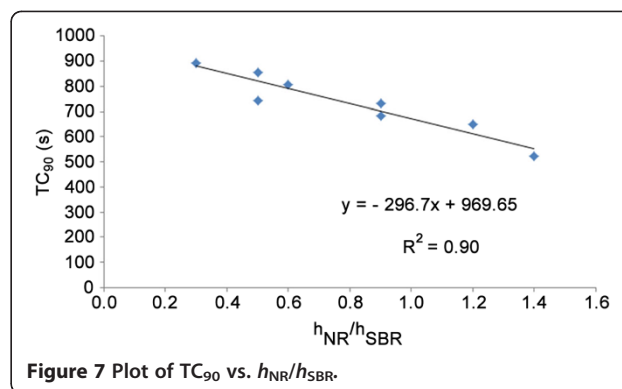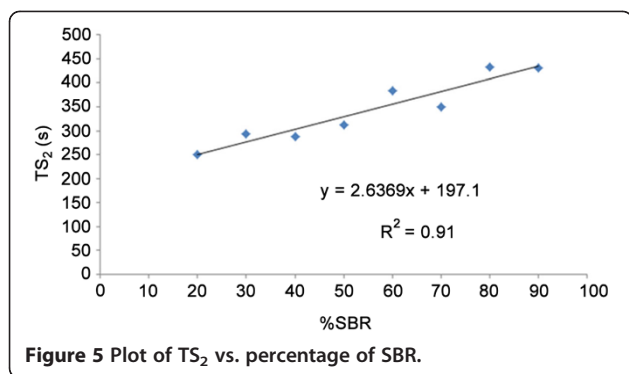
Equation 2). The correlation yielded a straight line with $R^2 = 0.90$ and 0.86. These can be expressed as follows:

$$Y = f(x)$$
$$(\text{normalized factor}) \text{ and } (h_{NR}/h_{SBR}) = f(\%SBR) \quad (1)$$

$$\text{Rheological properties} = f(\%SBR) \quad (2)$$

This approach suggests an acceptable relation between rheological properties of rubber blends based on NR/SBR with their thermal behavior, which can be represented by Equation 3 (Figures 7, 8, 9, 10, 11, 12) as

$$(\text{normalized factor}) \text{ and } (h_{NR}/h_{SBR})$$
$$= f(\text{rheological properties}) \quad (3)$$

It has been suggested that according to Equation 3, it is possible to determine the rheological nature of rubber blends based on their thermogravimetric analysis (normalized factor and h_{NR}/h_{SBR}). For example in this study, TC_{90} for the samples are related with h_{NR}/h_{SBR} and normalized factor as showed in Equations 4 and 5:

$$TC_{90} = -369.18x + 1010.6, R^2 = 0.95, \text{where } x$$
$$= h_{NR}/h_{SBR} \quad (4)$$

$$TC_{90} = -316.73x + 957.42, R^2 = 0.95, \text{where } x$$
$$= \text{normalized factor} \quad (5)$$

As shown in Figures 9 and 12, the relationship between CRI with h_{NR}/h_{SBR} and normalized factor is not linear and it has been fitted to a polynomial equation type. In this case the precision obtained using polynomial equation ($R^2 = 0.90$ and 0.93) is considerable.
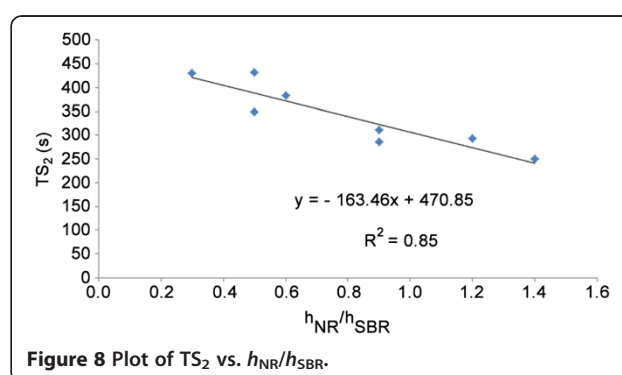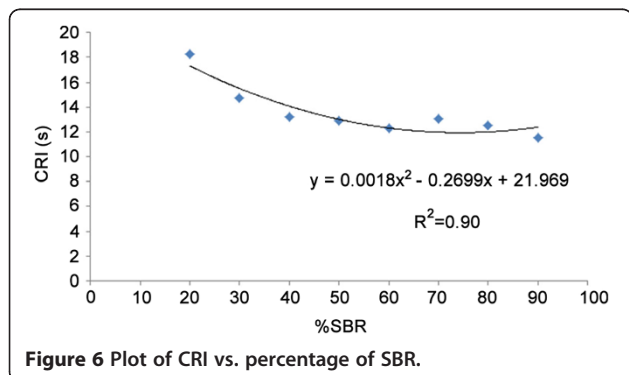
For those systems where the implemented process is analysis or any other method, the acceptance or rejection of measurements has nothing to do with tolerance percentages, but assessment can be done by following equation [19]:
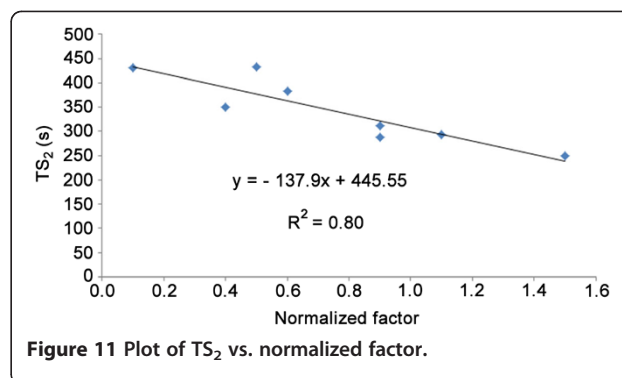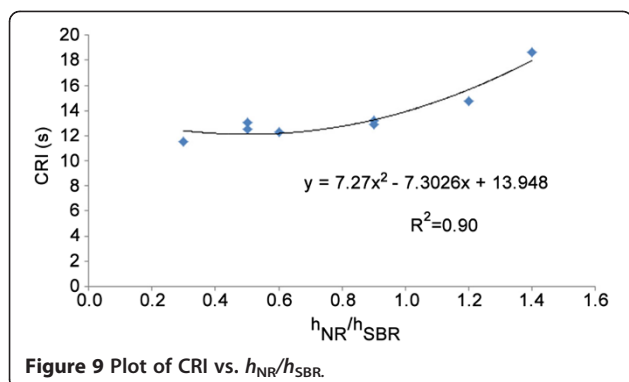
$$\%\text{relative error} = \frac{\text{experimental result–calculated result}}{\text{experimental result}} \times 100$$

It showed be noted that

– % Relative error fewer than 10% will be accepted.
– % Relative error from 10% to 30% may be accepted, but it depends on the process type.
– % Relative error more than 30% is not accepted.

During the measurements, a relative error of <10% were found for the TC_{90} and CRI, and <20% for the TS_2 calculated from the structure-properties modeling techniques (Figures 7, 8, 9, 10, 11, 12). The results shown in Tables 4, 5, 6, 7, 8, 9 suggest that the rheological properties of the blends can be predicted by two useful factors

Figure 6 Plot of CRI vs. percentage of SBR.

Figure 8 Plot of TS_2 vs. h_{NR}/h_{SBR}.

Figure 9 Plot of CRI vs. h_{NR}/h_{SBR}.

Figure 11 Plot of TS_2 vs. normalized factor.

and there is no significant difference between both calculated and experimental data. The accuracy and precision of the results have been statistically evaluated.

Because of the case study and repeatability of experiments, similar samples of BR_{20}, BR_{50}, and BR_{80} were prepared, and the rheological properties of these rubber materials were measured. The results obtained from both experimental and the structure-properties modeling techniques shown in Tables 10 and 11 are in good agreement. The accuracy and precision of the results have been statistically evaluated using 'SPSS' software (Tables 12, 13, 14, 15, 16, 17). In all cases, Pearson's correlation is more than 0.90 and the correlation is significant at the 0.05 level and indicative of the validity of the employed method.

Experimental
Sample preparation
Generally, base of tires compounds are unsaturated polymers that have been strengthened with carbon black and are vulcanized by sulfur system. Since any change in the formulations will influence the TG-DTG curves, all components of the samples were kept constant except the percentages of SBR in the NR/SBR blends. NR and SBR were mixed in varying compositions, between 0 and 100 phr. Samples containing weight fractions of SBR are shown as SBR_{20} - SBR_{30} - SBR_{40} - SBR_{50} - SBR_{60} - SBR_{70} - SBR_{80} -

SBR_{90} - SBR_{100}. The formulated compounds were mixed on a two-roll mill according to the standard procedure. During mixture, procedure time and cutting operation have been checked. The temperature range for mixing was 70°C to 75°C. The order and average time for mixing were as follows:

- 15 min, mastication;
- 25 min, addition of filler;
- 10 min, addition of powdered materials; and
- 15 min, addition of curatives (mill temperature at 65°C).

Thermogravimetric analysis
Thermogravimetric analysis of NR/SBR blends was carried out for uncured sample. The heating rate was kept at 20°C/min and continued from ambient temperature to 700°C. In all analysis, a nominal 15-mg uncured sample was used. Under nitrogen atmosphere, the samples were heated from 30°C up to 550°C to monitor the weight loss of volatile components. At 550°C, the gas flow was changed from nitrogen to air (flow rate of 10 mL/min) and heating continued until constant weight loss was achieved at 700°C. Weight loss of the sample was continuously measured as a function of the temperature. The total analysis time was approximately 35 min.

Figure 10 Plot of TC_{90} vs. normalized factor.

Figure 12 Plot of CRI vs. normalized factor.
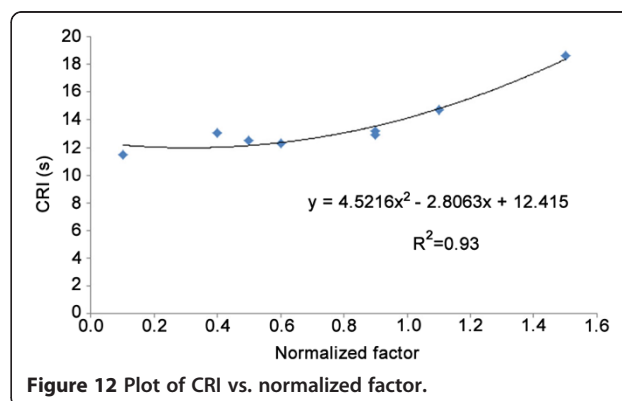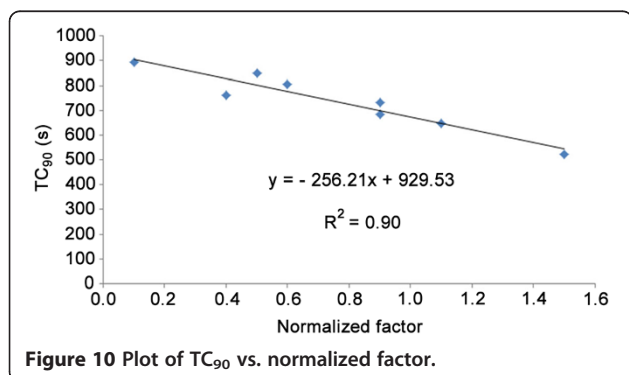
Table 4 Comparison of measured and calculated TC$_{90}$ to peak height ratio

h_{NR}/h_{SBR} value	Measured TC$_{90}$ (s)	Calculated TC$_{90}$[a] (s)	Error (%)
1.4	482	493.7	2.3
1.2	555	567.1	2.1
0.9	684	678.5	0.8
0.9	733	678.5	7.4
0.6	754	789.1	4.6
0.5	858	826.0	3.7
0.5	787	826.0	4.9
0.3	906	899.8	0.7

[a]Curve fitting equation, TC$_{90}$ = −369.18x + 1,010.6, R^2 = 0.95, where x = h$_{NR}$/h$_{SBR}$.

Table 7 Comparison of measured and calculated TC$_{90}$ by correlating TC$_{90}$ to normalized factor

Normalized factor	Measured TC$_{90}$ (s)	Calculated TC$_{90}$[a] (s)	Error (%)
1.5	482	482.3	0.06
1.1	555	609.4	9.80
0.9	684	672.3	1.70
0.9	733	672.3	8.20
0.6	754	767.3	1.70
0.4	858	830.7	3.10
0.5	787	799.0	1.50
0.1	906	925.7	2.10

[a]Curve fitting equation, TC$_{90}$ = −316.73x + 957.42, R^2 = 0.95, where x = normalized factor.

Table 5 Comparison of measured and calculated CRI to peak height ratio

h_{NR}/h_{SBR} value	Measured CRI (s^{-1})	Calculated CRI[a] (s^{-1})	Error (%)
1.4	18.657	17.975	3.65
1.2	14.717	15.656	6.38
0.9	13.236	13.264	0.21
0.9	12.917	13.264	2.69
0.6	12.293	12.184	0.89
0.5	13.097	12.114	7.50
0.5	12.547	12.114	3.45
0.3	11.514	12.412	7.80

[a]Curve fitting equation, CRI = 7.2700x^2 − 7.3026x + 13.9480, R^2 = 0.90, where x = h$_{NR}$/h$_{SBR}$.

Table 8 Comparison of measured and calculated CRI by correlating CRI to normalized factor

Normalized factor	Measured CRI (s^{-1})	Calculated CRI[a] (s^{-1})	Error (%)
1.5	18.657	18.294	1.94
1.1	14.717	14.799	0.56
0.9	13.236	13.552	2.39
0.9	12.917	13.552	4.92
0.6	12.293	12.359	0.54
0.4	13.097	12.016	8.25
0.5	12.547	12.142	3.28
0.1	11.514	12.179	5.77

[a]Curve fitting equation, CRI = 4.5216x^2 − 2.8063x + 12.4150, R^2 = 0.93, where x = normalized factor.

Table 6 Comparison of measured and calculated TS$_2$ to peak height ratio

h_{NR}/h_{SBR} value	Measured TS$_2$ (s)	Calculated TS$_2$[a] (s)	Error (%)
1.4	246	248.6	0.8
1.2	272	261.3	3.9
0.9	268	297.2	10.8
0.9	307	297.2	3.1
0.6	374	353.2	5.5
0.5	320	376.4	17.6
0.5	427	376.4	11.8
0.3	426	429.4	0.7

[a]Curve fitting equation, TS$_2$ = 111.93x^2 − 354.6x + 525.72, R^2 = 0.80, where x = h$_{NR}$/h$_{SBR}$.

Table 9 Comparison of measured and calculated TS$_2$ by correlating TS$_2$ to normalized factor

Normalized factor	Measured TS$_2$ (s)	Calculated TS$_2$[a] (s)	Error (%)
1.5	246	237.0	3.6
1.1	272	279.7	2.8
0.9	268	304.6	13.6
0.9	307	304.6	0.7
0.6	374	346.5	7.3
0.4	320	377.4	17.9
0.5	427	361.6	15.3
0.1	426	428.3	0.5

[a]Curve fitting equation, TS$_2$ = 29.968x^2 − 184.54x + 46.46, R^2 = 0.80, where x = normalized factor.

Table 10 Results of the case study using h_{NR}/h_{BR}

SBR (%)	Measured TS_2 (s)	Calculated TS_2 (s)	Error (%)	Measured TC_{90} (s)	Calculated TC_{90} (s)	Error (%)	Measured CRI (s^{-1})	Calculated CRI (s^{-1})	Error (%)
20	240	248.6	3.5	479	493.7	3.1	18.901	17.975	4.90
50	312	297.2	4.7	728	678.5	6.8	13.508	13.264	1.81
80	416	376.4	9.5	795	826	3.9	11.898	12.114	1.81

Table 11 Results of the case study using normalized factor

SBR (%)	Measured TS_2 (s)	Calculated TS_2 (s)	Error (%)	Measured TC_{90} (s)	Calculated TC_{90} (s)	Error (%)	Measured CRI (s^{-1})	Calculated CRI (s^{-1})	Error (%)
20	240	237.0	1.3	479	482.3	0.7	18.901	18.294	3.21
50	312	304.6	2.4	728	672.3	7.7	13.508	13.552	0.32
80	416	361.6	13.0	795	799.0	0.5	11.898	12.142	2.05

Table 12 Statistical results of the comparison of experimental and calculated TS_2 using h_{NR}/h_{BR}

	Pearson's correlation	Sig (1-tailed)	Number
Measured TS_2	1	-	3
Calculated TS_2	0.999[a]	0.010	3

[a]Correlation is significant at the 0.05 level (1-tailed). Sig, significance.

Table 15 Statistical results of the comparison of experimental and calculated TS2 using normalized factor

	Pearson's correlation	Sig (1-tailed)	Number
Measured TS_2	1	-	3
Calculated TS_2	0.988[a]	0.041	3

[a]Correlation is significant at the 0.05 level (1-tailed). Sig, significance.

Table 13 Statistical results of the comparison of experimental and calculated TC_{90} using h_{NR}/h_{BR}

	Pearson's correlation	Sig (1-tailed)	Number
Measured TC_{90}	1	-	3
Calculated TC_{90}	0.981[a]	0.050	3

[a]Correlation is significant at the 0.05 level (1-tailed). significance.

Table 16 Statistical results of the comparison of experimental and calculated TC_{90} using normalized factor

	Pearson's correlation	Sig (1-tailed)	Number
Measured TC_{90}	1	-	3
Calculated TC_{90}	0.981[a]	0.050	3

[a]Correlation is significant at the 0.05 level (1-tailed). significance.

Table 14 Statistical results of the comparison of experimental and calculated CRI using h_{NR}/h_{BR}

	Pearson's correlation	Sig (1-tailed)	Number
Measured CRI	1	-	3
Calculated CRI	0.986[a]	0.043	3

[a]Correlation is significant at the 0.05 level (1-tailed). significance.

Table 17 Statistical results of comparison of experimental and calculated CRI using normalized factor

	Pearson's correlation	Sig (1-tailed)	Number
Measured CRI	1	-	3
Calculated CRI	0.984[a]	0.044	3

[a]Correlation is significant at the 0.05 level (1-tailed). significance.

Conclusions

From the present study of rubber blends based on NR/SBR, a novel approach is reported to quantify rheological properties of rubber compounds using the structure-properties modeling techniques [calibration curve obtained by correlating the factors obtained by TGA-DTG graphs (h_{NR}/h_{SBR} – normalized factor) and rheological nature of samples]. It is well known that the degradation of SBR takes place at a higher temperature than that of NR. However, blends show a higher degradation temperature than NR.

From the obtained results it can be concluded that in rubber compounds based on NR/SBR blends, when all components of the samples were kept constant except the percentages of SBR in the blends, the relation between factors obtained by TG-DTG profiles and rheological properties of rubber compounds has been fitted to a linear and polynomial equation. It is possible to predict rheological properties of blends using these equations by TG-DTG method by comparing real rheological properties of rubber compounds to those predicted by both calibration curves to test the accuracy of the thermogravimetric method. The study confirms the accuracy of TG-DTG technique for rapid prediction of rheological properties of rubber blends.

The precision of this method greatly depends on the starting material, condition of sample preparation, and rheological test methods, which for all samples should be the same.

Authors' information
FM is a Ph.D. in applied chemistry. She is an assistant professor in the Applied Chemistry Department at Islamic Azad University, Tehran North Branch. After her Ph.D., she started her carrier in rubber compounding and technology, synthesis of new ingredients for rubber industries, optimization of aging conditions of rubber compound, scrap tires management, and the structure-properties modeling of rubber compounds. STG is a Ph.D. in organic chemistry. He is the head of Iran Rubber Research Institute, associate professor of Organic Chemistry Department at Islamic Azad University, Tehran North Branch. He has interest in rubber compounding and technology, synthesis of new ingredients for rubber industries, application of nanoparticles in rubber compounds, and scrap tires management. He presented his research work in many conferences and also published papers in peer-reviewed international journals. MM is also a Ph.D. in applied chemistry. She is an assistant professor of applied chemistry department at Islamic Azad University, Tehran North Branch. She has also interest in rubber compounding and technology, synthesis of new ingredients for rubber industries, application of nanoparticles in rubber compounds, scrap tires management, and water treatment.

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