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Determination of the crosslink density of conductive ternary rubber vulcanizates by solvent penetration

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The order and time period of mixing were as follows:

0–3 min: mastication of NR,

3–6 min: addition and Mastication of the other two rubbers.

6–9 min: addition of 1/3 filler plus 1/3 oil.

9–14 min: addition of 1/3 filler plus 1/3 oil.

14—20 min: addition of remaining filler and oil,

20–29 min: addition of other ingredients.

29–30 min: refining through tight nip gap and dump (the nip gap was 1.5 mm).

After mixing the rubber compositions were molded in an electrically heated hydraulic press to the optimum cure (90% of the maximum cure) using molding conditions previously determined from the torque data obtained by a Monsanto rheometer (R-100).

2.3. Swelling measurements

Rubber blends were weighed and inserted in test tubes containing solvents with different cohesive energy densities (c.e.d) (as tabulated in Table 2) in an oven at 30°C. The rubber blends were removed from the solvent and blotted with filter paper to remove excess solvent on the sample surface. The rubber blends were then weighed to an accuracy of 0.1 mg (using digital balance) at a given time and a fixed temperature (30°C).

With an assumption of volume additivity of the polymer and absorbed solvent in swelling, the volume swelling ratio, defined as the reciprocal of the polymer volume fraction in a swollen gel, was calculated using the following equation:

$$\frac{1}{\Phi_p} = 1 + \frac{W_s}{W_p}\frac{\rho_p}{\rho_s}, \qquad (1)$$

where W_p and W_s are the weights of the dried polymer (rubber) and absorbed solvent, respectively, and ρ_p and ρ_s are the densities of the dried polymer and solvent, respectively.

3. Results and discussion

A crosslinked elastomer hardly dissolves in a solvent. Dispersion is resisted because the crosslinks restrict the movement and complete separation of the chains, but the elastomer does swell when the solvent molecules diffuse into the network and cause the chains to expand. This expansion is counteracted by the tendency for the chains to coil up and eventually an equilibrium degree of swelling is established which depends on the solvent and the crosslink density, i.e. the higher the crosslink density the lower the swelling [11].

3.1. Penetration of solvents into the conductive ternary rubber vulcanizates

From a practical point of view it is very important to know the capacity of crosslinked polymers for swelling in various liquid and vapour media. This capacity is assessed by the degree of swelling, expressed as the amount of liquid sorbed by a polymer.

The swelling of rubber involves a diffusion process by which the liquid is transported from one part of the sample to another. This diffusion theory in elastomers [12,13] is based on the assumption that the swelling commences by the sorption of the liquid in the surface of the sample to a certain concentration equal to that of the whole sample at final equilibrium; then the swelling proceeds by increasing the depth of the swollen layers at a penetration rate, P, [14,15]:

$$P = \frac{1}{2}\frac{\mathrm{d}(M_t/M_e)}{\mathrm{d}(t^{1/2}/S)}, \qquad (2)$$

for a rubber sheet of thickness S. M_e and M_t are the weight uptake of the liquid at equilibrium and after time t, respectively. The diffusion of a liquid in the bulk of the rubber depends on the homogeneity of the mix and the way that the carbon is aggregated [16].

The relation between the average diffusion coefficient, D, and the penetration rate, P, is given as [16]:

$$D = \pi P^2/4. \qquad (3)$$

The ternary rubber vulcanizates were subjected to repeated measurements to discuss the influence of changing the IIR or/and SBR content on the penetration rate of both kerosene and benzene in these rubber matrices.

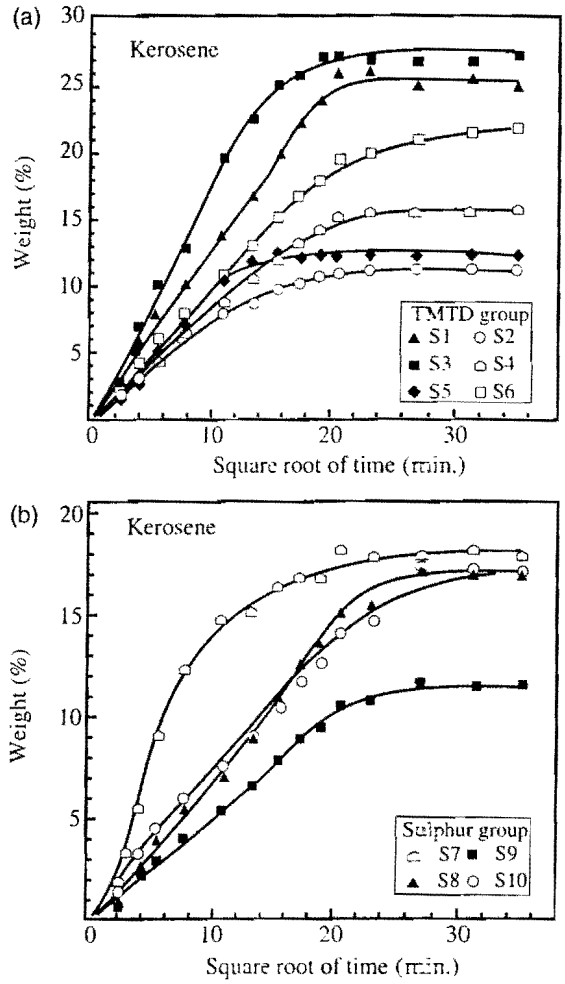

Fig. 1. Percentage weight swelling versus square root of time for samples (in kerosene): (a) in group A, (b) in group B.

The percentage increase in weight due to swelling (in kerosene and benzene respectively) is plotted against the square root of time, in minutes (cf. Figs. 1 and 2)). The penetration curves are similar in character. The slope of the straight portions obtained at the early part of the curves was calculated. This slope is equal to $M_t/t^{1/2}$, and at the equilibrium volume swelling, M_e values were obtained. Using Eqs. (2) and (3) the penetration rate and consequently the diffusion coefficient were calculated.

Fig. 3a and 3b are plots of $\Phi_p/(1-\Phi_p)$ versus the penetration rate P for both vulcanizing agents sulphur and TMTD, where Φ_p is the volume content of IIR rubber in the mix.

The two vulcanizing agents have relatively opposite effects on the penetration rate of both kerosene and benzene through these ternary rubber mixes. In the case of sulphur the solvent penetration may be attributed to the slight increase in crosslinking density and aggregation of HAF black inside the bulk rubber mix. These aggregation and crosslinking happen at different plate lamellae which act as screens, delaying the penetration rate of the solvent (kerosene and benzene) molecules into the bulk of the rubber mix. Rubber reinforcement due to the addition of carbon black causes restriction to the equilibrium volume swelling. At high IIR content (> 30 phr), the volume fraction of the polymer (SBR) in the mix decreases, causing an increase in the penetration rate of the solvent.

The opposite behavior is detected and can be explained on the basis of the above discussion for the case of samples filled with TMTD vulcanizing agent.

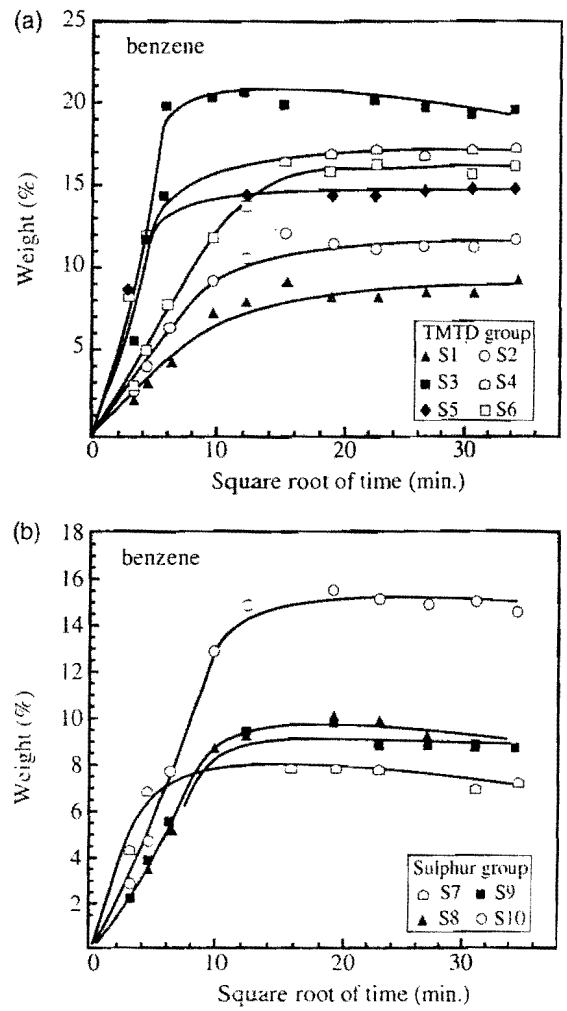

Fig. 2. Percentage weight swelling versus square root of time for samples (in benzene): (a) in group A, (b) in group B.

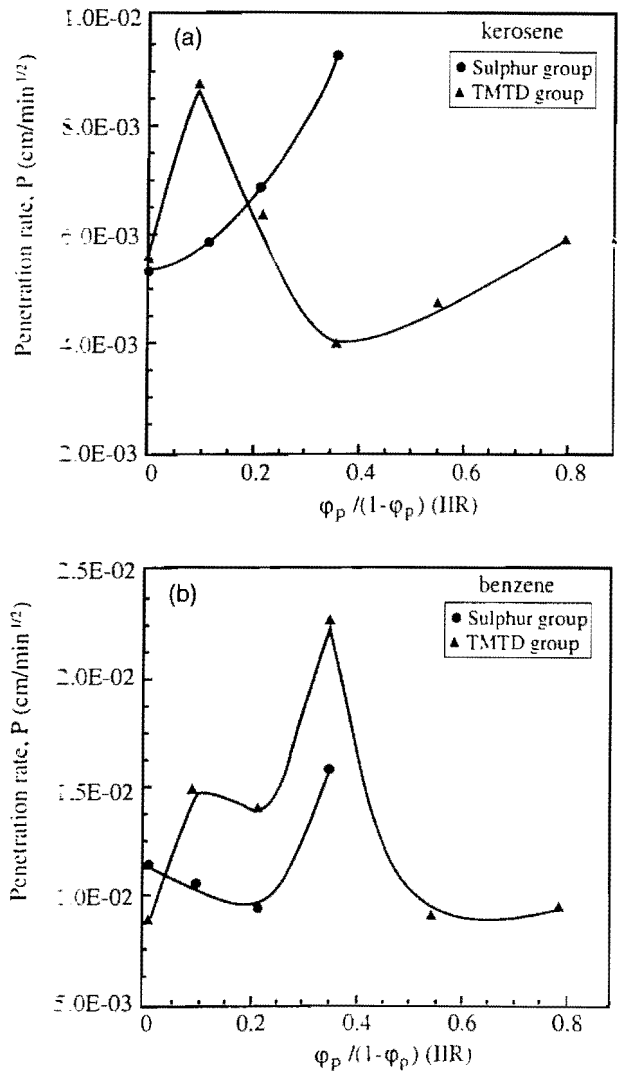

Fig. 3. Penetration rate versus $\Phi_p/(1-\Phi_p)$; Φ_p is the volume fraction of IIR in the mix for group A and group B: (a) in kerosene, (b) in benzene.

3.2. *Determination of the crosslinking density of ternary rubber vulcanizates by swelling*

One of the most important structural parameters characterizing crosslinked polymer is $\bar{M}_c$ the average molecular weight between crosslinks, which is directly related to the crosslink density. The magnitude of $\bar{M}_c$ significantly affects the physical and mechanical properties of crosslinked polymers and its determination has a great practical significance. Equilibrium swelling is widely used to determine $\bar{M}_c$ [5,17–20]. Early research by Flory and Rehner [4,5] laid the foundations for the analysis of the equilibrium swelling. According to the theory of Flory and Rehner [21], for a perfect network,

$$\bar{M}_c = -V_1 \rho_p \frac{\Phi_p^{1/3} - \Phi_p^{1/2}}{\ln(1-\Phi_p) + \Phi_p + \chi_1 \Phi_p^2}, \qquad (4)$$

where $\bar{M}_c$ is the average molecular weight of the polymer between crosslinks, V_1 is the molar volume of the solvent, ρ_p is the polymer density, Φ_p is the volume fraction of the polymer in the swollen matrix and χ_1 is the Flory–Huggins interaction parameter between solvent and polymer.

The swelling ratio, s, is equal to $1/\Phi_p$. Here, the crosslink density, υ_e, defined by some authors [22] as the number of elastically effective chains, totally included in a perfect network, per unit volume is simply equal to

$$\nu_e = \frac{\rho_p N}{\bar{M}_c}, \qquad (5)$$

where N is Avogadro's number.

In most cases, especially for determining the crosslink density of vulcanized rubbers, a method due to Gee [23] is used. This method is based on the assumption of maximum swelling of the vulcanizate in a liquid whose cohesive energy density c.e.d. (δ) equals that of the vulcanizate.

After experimentally determining the degree of swelling of all ternary rubbers used in different solvents (with different c.e.d.), the equilibrium degree of swelling is plotted against the c.e.d. of the liquids. Fig. 4a and 4b represent examples for this relation for sample S_3 and S_7 (sulphur and TMTD group respectively). The result for all liquids is a single master curve, having a maximum at the abscissa corresponding to the c.e.d. of the polymer. The parameter χ_1 is then calculated by substituting this quantity into the equation:

$$\chi_1 = \frac{(\delta_1 - \delta_2)^2 V_1}{RT}, \qquad (6)$$

where δ_1 and δ_2 are the solubility parameter or the cohesive energy density for the solvent and polymer respectively. V_1 is the partial molar volume of the solvent and R is the universal gas constant.

It is clearly observed from Fig. 4a and 4b that the c.e.d. values of rubber samples change according to the type of the vulcanizing agent used (= 8.6 and = 9.3 for sulphur and TMTD group respectively).

Substituting the value of χ_1 for each group (for the case of kerosene and benzene) in Eq. (4) one can calculate the value of M_c for each sample and hence using Eq. (5) the crosslink density υ_e is deduced.

It should, however, be pointed out that this method for determination of χ_1 is inaccurate. The polymer–solvent interaction parameter was estimated using the following formula by Noda et al. [24] and Brandrup et al. [25] for the case of polystyrene system (at equilibrium):

$$\chi_1 = 0.431 - 0.311\Phi_p - 0.036\Phi_p^2. \qquad (7)$$

By applying this equation to our case, the crosslink density varies as is shown in Fig. 5 which represents the variation of υ_e (for group B) with the IIR content.

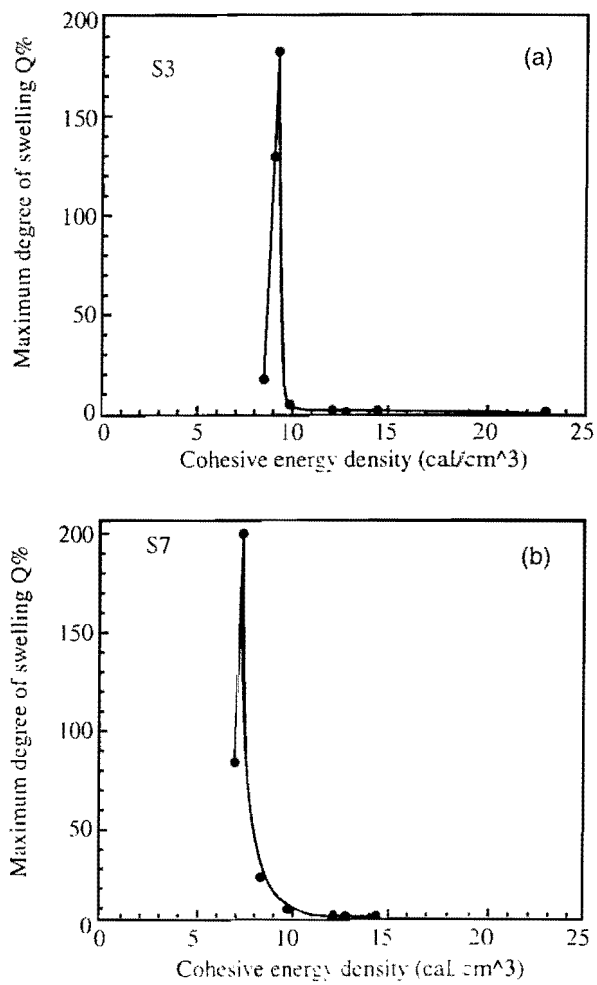

Fig. 4. The equilibrium (maximum) degree of swelling versus the cohesive energy density of liquids: (a) sample S_3, (b) sample S_7.

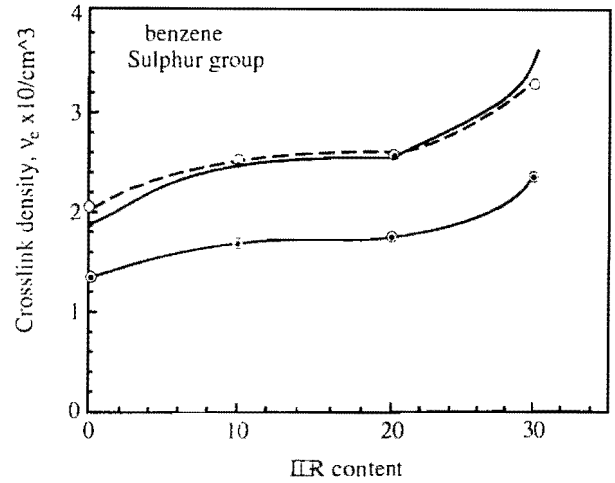

Fig. 5. Variation of the crosslink density υ_e with the IIR content for group B: (●) experimental values, (⊖) calculated values using Eq. (7), (○) calculated values using the modified equation (8).

A small discrepancy between the theoretically estimated values of υ_e and the experimentally determined one is obtained. By adding the fourth term of the polynomial (in Φ_p) of Eq. (7), χ_1 becomes:

$$\chi_1 = 0.431 - 0.311\Phi_p - 0.036\Phi_p^2 - 1.8\Phi_p^3 \qquad (8)$$

and a fairly good agreement of practical and theoretical calculated values of υ_e (in kerosene) for all samples is obtained.

4. Conclusions

From the above study one may conclude that:

(1) The penetration rate of benzene in ternary rubber vulcanizates (for group A) decreases with an increase of IIR content up to 30 phr. For concentrations >30 phr there is a remarkable increase in penetration rates.

(2) The penetration rate of kerosene increases with an increase of IRR content (for samples of group B) up to 30 phr.

(3) The penetration rate P of benzene is greater than that detected for kerosene for all composites.

(4) The cohesive energy density δ calculated using the Gee method was changed with the vulcanizing agent ($\delta = 8.6$ for sulphur and $\delta = 9.3$ for TMTD).

(5) The crosslink density is higher for sulphur group than for TMTD group.

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