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CROSSLINKING WITH S_2Cl_2 OF EPDM RUBBERS AND OF EPDM GRAFTED WITH POLYACRYLIC ACID

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Abstract—EPDM rubber swollen in n-butylacetate was crosslinked with S_2Cl_2 solution in n-butylacetate. From swelling measurements in benzene and the Flory–Rehner equation, the density of elastically-active network chains was calculated as a function of time. Kinetics have been discussed; the diffusion process of S_2Cl_2 in the swollen polymer plays a very important role. Crosslinking of EPDM grafted with polyacrylic acid has been carried out on thin films using S_2Cl_2 as reagent and n-butylacetate as swelling agent, showing similar kinetic behaviour. Intermolecular bonds are due both to sulphur bridges in EPDM and to sulphur and to hydrogen bonds in EPDM grafted samples.

INTRODUCTION

Saturated ethylene–propylene copolymer (EPR) is generally crosslinked in the presence of peroxides as crosslinking agents, but the technology is difficult. Terpolymers have long been prepared starting from ethylene–propylene, and also a doubly unsaturated monomer (EPDM) so that they may be vulcanized at high temperatures by traditional methods based on sulphur.

The possibility of crosslinking EPDM at room temperature with sulphur chloride is described in this paper, as already known e.g. for natural rubber and for polybutadiene [1–3]. Since rubbers modified by the introduction of polar groups are of practical importance, we deemed it interesting to study also the crosslinking with S_2Cl_2 of EPDM grafted with acrylic acid. Grafting of saturated EPR polymers was first studied at this Institute [4].

Grafted EPDM may be crosslinked since not all double bonds of the elastomeric chain are removed by grafting. As a matter of fact, grafting was shown [5] to occur predominantly at the α position with respect to the double bond.

The method adopted by us allows the preparation and vulcanization at room temperature of a thin crosslinked film but it is not suitable for the vulcanization of quite thick films.

EXPERIMENTAL

Elastomer purification

EPDM (Montedison), having the following composition: ethylene 57·8 mole %, propylene 41·2 mole %, 5-ethyliden-2-norbornene 1 mole %, was purified by dissolution in toluene and subsequent precipitation in methanol. Its Mooney viscosity was 40–50 and its density 0·865 g/cm^3.

Crosslinking

Crosslinking was carried out with S_2Cl_2 at room temperature (*ca.* 25°). S_2Cl_2 (BDH) was used because it is the stablest of the sulphur chlorides. The reagent is quite reactive so that, if used alone, it causes considerable crosslinking on the surface before diffusing to the inside. Therefore, dilution of the reagent with a rubber-swelling solvent is convenient both to decrease the crosslinking rate and to favour the diffusion of the reagent. The solvent must neither react with S_2Cl_2 [6] nor dissolve the polymer. The order of increasing power for EPDM swelling by the solvents shown in Table 1 is nitromethane–methylethylketone–n-butylacetate; this last-named was selected as the diluent for S_2Cl_2.

The elastomer was compression moulded at 170–175° at 50 kg/cm^2 for 5 min. Small plates, 2 mm thick, were obtained; from them, square-based small prisms of 0·3–0·4 g weight were cut. These test-pieces were swollen for 24 hr in n-butylacetate and then placed in a S_2Cl_2 solution in n-butylacetate at a concentration ranging from 2 to 6 g/l; its volume was such that the molar ratios between S_2Cl_2 and the double bonds were as fixed in advance, by operating in the absence of dampness.

After crosslinking, the test-pieces were washed in n-butylacetate, and then dried at 50° at reduced pressure to constant weight. The degree of swelling q_m in benzene was then measured in duplicate.

During crosslinking, the test-piece increases in weight, since S_2Cl_2 is chemically bound to rubber. In crosslinked products, both chlorine and sulphur are present, as shown

Table 1. Tests of swelling of uncrosslinked EPDM at 25°

Solvent	Degree of swelling q_m 24 hr	25 days
Nitromethane	1·005	—
Methylethylketone	1·135	1·164
n-Butylacetate	1·535	1·577

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by elemental analysis. During the subsequent swelling in benzene, the rubber weight often decreases, the more the less is the crosslinking, because of solution of the non-crosslinked material.

Reaction of grafting

As an example, we report the procedure adopted for obtaining the sample employed in the research referred to in Fig. 3. Grafted EPDM was prepared by dissolving the purified terpolymer (20 g) in toluene (600 cm^3) at 80°. Under inert atmosphere (nitrogen), dicumylperoxide (0·1 g) and freshly distilled acrylic acid (10 cm^3) were added. Reaction was allowed to occur for 10 hr. Part of the solution was precipitated for analysis. The reaction crude product contains 33 per cent polyacrylic acid (of which 60 per cent is grafted and 40 per cent is homopolymer, as found by Kumagawa extraction with boiling methanol). Furthermore, not all EPDM chains are grafted (hexane extract corresponds to 15 per cent of crude).

The crude solution, concentrated under reduced pressure to give a polymer content of 7 per cent, was placed in the form of films on microscope slides (*ca.* 20 cm^2 area) and then dried. The film thickness (30–180 μm) was regulated by varying the amount of solution employed. Use of a film of grafted crude product was preferred over that of grafted product, isolated by fractionation, it being difficult to dissolve the latter after fractionation.

Crosslinking of grafted EPDM

Grafted EPDM films, without being detached from the slides, were swollen in n-butylacetate for 1·5 hr when swelling reaches a limiting value even for the thickest films.

The films, always on the slides and swollen in n-butylacetate, were immersed in the S_2Cl_2 solution in n-butylacetate for the desired time. After crosslinking, it was easier to detach films from the slides for the more highly crosslinked samples. The films, washed in n-butylacetate and dried, were subsequently swollen in benzene in order to measure q_m. We found no appreciable swelling in benzene of polyacrylic acid homopolymer. Therefore it is right to attribute swelling of the grafted product to the rubber fraction only. The kinetics of swelling for films with different degree of crosslinking (reaction times of 1·25 and 1·50 hr) showed that the values of q_m are nearly constant after 24 hr. Therefore we chose a 24-hr contact time for all runs.

Measurements of the degree of swelling

It is known [7] that the degree of swelling q_m in a solvent of a crosslinked polymer is the ratio of the swollen gel volume V_t to the crosslinked polymer volume V_r:

$$q_m = \frac{V_t}{V_r} \qquad (1)$$

$V_t = V_0 + V_r$, where V_0 is the volume of the swelling liquid. After solvent evaporation, rubber is left; thus $V_r = P_r/\rho_r$; furthermore: $V_0 = (P_t - P_r)/\rho_0$ where P and ρ are weight and density; the indices are as defined above.

The experimental method adopted for V_t and V_r measurement was that of Crespi and Bruzzone [8].

For grafted EPDM, the volumes of polyacrylic acid and of rubber were considered as additive for the calculation of q_m [9] and swelling in benzene was attributed only to the rubber fraction, as already done by other authors [10].

Measurement of the crosslinking density

The simplest method for evaluation of the number of elastically effective chains per unit volume of rubber is that based on swelling measurements according to the well known semi-empirical Flory–Rehner equation [11,12]:

$$\log(1 - v_r) + v_r + \mu v_r^2 = -\nu V_1(v_r^{1/3} - v_r/2), \qquad (2)$$

where $v_r = 1/q_m$ is the volume fraction of rubber in the swollen material at equilibrium: μ is the Huggins solvent–polymer interaction coefficient, V_1 is the solvent molar volume and $\nu = \rho_r/\bar{M}_c$ is the number of elastically effective chains per unit volume of rubber ($\bar{M}_c$ is the molecular weight of the chain portion between the two crosslinks). In the case of EPDM, several empirical relationships connect parameter μ to v_r [8, 10, 14–16]. We chose that of Zamboni *et al.* [15].

$$\mu = 0{\cdot}477 + 0{\cdot}303\, v_r, \qquad (3)$$

since it refers to an EPDM having an almost identical composition with that of the terpolymer used by us and it refers to benzene as the solvent.

We set up a computing programme for a rapid calculation of the crosslinking density from the experimental data on q_m.

Also in the case of grafted EPDM, the density of the elastically effective chains has been calculated by Eqns. (2) and (3), starting from the values of q_m referred to the elastomeric fraction only. In fact, the relationship between ν and v_r exclusively depend on the type of rubber and on the solvent which has, in our case, no interaction with polyacrylic acid.

Limits of crosslinking

For the EPDM used, the weight of the rubber chain portions between the two unsaturations on average is:

$$\bar{M}_c = \frac{120}{3{\cdot}5}\, 100 = 3430$$

where 120 is the molecular weight of the 5-ethylene-2-norbornene unit, which is present in EPDM at 3·5 wt % (= 1 mole %).

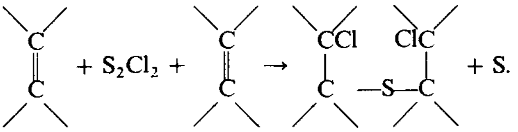
If crosslinking were complete, the limiting crosslinking density should be

$$\nu_{(\text{theoretical})} = \frac{\rho_r}{\bar{M}_c} = 25{\cdot}2 \times 10^{-5}\ \text{mol/cm}^3. \qquad (5)$$

Since two elastically effective chains are required for each crosslink the highest weight increase of EPDM for a complete reaction with S_2Cl_2 [see Eqn. (6)] should be 1·97 per cent. In the calculation of v_r, we neglected the very low variation of rubber density following the occurrence of crosslinking; use was made of $\rho_r = 0{\cdot}865$ g/cm^3.

RESULTS AND DISCUSSION

Although crosslinking of rubber double bonds with S_2Cl_2 has been known for more than a century [1], its mechanism has not yet been established. By analogy with what has been stated by other authors for natural rubber [17, 18], crosslinking of EPDM can be assumed to be:



(6)

This is also suggested by Blow [19]. Other schemes are possible [20] involving two reaction steps. There could be formation of an intermediate between an elastomeric chain and S_2Cl_2; then the intermediate could react with a second elastomeric chain. Finally, the elastomeric chains could be bound by a —S—S— bond instead of a —S— bond.

Elemental analysis for chlorine and sulphur in crosslinked EPDM samples did not allow us to establish easily which reaction has actually occurred because of the small amount of double bonds present in EPDM and the analytical errors. In any case, the calculation of ν, as done in our work, is not influenced by the presence of a —S—S— bond instead of a —S— bond. All authors admit the consumption of one S_2Cl_2 mole for every pair of bonded double bonds, i.e. for two elastically effective chains.

The same mechanism of crosslinking can be assumed in the case of the grafted EPDM. S_2Cl_2, under our conditions, is reactive only with double bonds. By treating polyacrylic acid at room temperature for 24 hr with S_2Cl_2 (both pure and diluted with n-butylacetate) no band shift has been found by i.r.; the spectrum is unaffected. Had —COCl or anhydride groups been formed, the vibration frequency of carbonyl would decrease. Furthermore, after treatment with S_2Cl_2, the polyacid is still perfectly soluble.

Previous kinetic studies on natural rubber in dilute solution [18] demonstrated that the order of reaction is one with respect to S_2Cl_2. In the case examined by us, owing to the EPDM copolymer's insolubility (even if swollen) in the solvent for the reagent S_2Cl_2, diffusion phenomena may be of great importance. Were diffusion the slowest process, the Fick law should be valid for reagent S_2Cl_2:

$$T = -D \operatorname{grad} C. \qquad (7)$$

By assuming that on the active centres C = 0, the equation becomes

$$T = \frac{C_{\text{ext}}}{R} \qquad (8)$$

where R is the overall resistance opposite to flux (g moles/cm^2 sec) and C_{ext} is the external concentration. A sample material balance indicates that

$$T = \frac{1}{2}\frac{V}{A}\frac{d\nu}{dt}, \qquad (9)$$

where V and A, respectively, are the rubber volume and its surface.

Instead, if the chemical reaction were the slowest, it may be assumed that it is first order with respect to S_2Cl_2 as well as with respect to the double bonds; since the reaction is stepwise [20] and involves one double bond for each step, then

$$\frac{d\nu}{dt} = K(2T - \nu)(S - \nu) \qquad (10)$$

the integral of which is

$$\ln \frac{2T - \nu}{S - \nu} = (2T - S)Kt + \text{Const.} \qquad (11)$$

where T is the number of S_2Cl_2 moles introduced into the system divided by rubber volume; S is the number of moles of double bond per cm^3 of rubber (= $25{\cdot}2 \times 10^{-5}$ moles/cm^3) and K is the kinetic constant multiplied by the ratio between the volumes of rubber and of the (rubber + solution) system, respectively.

EPDM crosslinkings reported in Fig. 1 were carried out by varying the initial concentration of S_2Cl_2 (2 g, 4 g and 6 g S_2Cl_2 per litre of n-butylacetate) and by keeping the molar ratio S_2Cl_2/double bonds constant at 6·28.

The figure shows the moles of the elastically effective chains per cm^3 of rubber (ν) vs the solution contact time: S-shaped curves may be observed. Acceleration is briefer the higher is the S_2Cl_2 concentration: this indicates that the kinetics of the process are influenced by diffusion. Then there is a stationary state followed by deceleration.

For steady diffusion, the initial ratio between solution and rubber may be important, since the concentration of S_2Cl_2 changes during the reaction. With the same concentration in the S_2Cl_2 solution (in this case 4 g/l), several runs were carried out in the presence

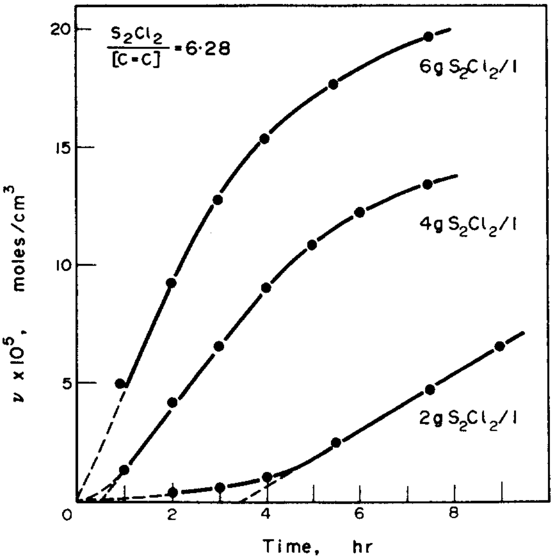


Fig. 1. Density of the elastically effective chains (ν) vs time for crosslinking of EPDM at different S_2Cl_2 concentrations. S_2Cl_2/double bonds molar ratio = 6·28.

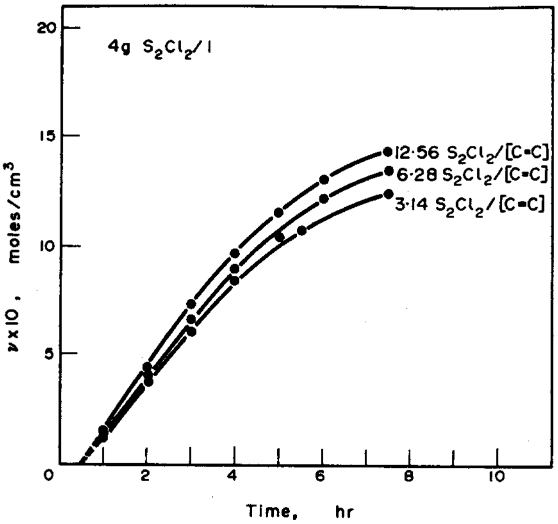


Fig. 2. Density of the elastically effective chains (ν) vs time for crosslinking of EPDM at the same S_2Cl_2 concentration (4 g/l), but at different S_2Cl_2/double bonds molar ratio.

of different amounts of solution so that ratios of 12·56:1; 6·28:1 and 3·14:1 between the numbers of moles of S_2Cl_2 and double bonds are obtained. Reactions are compared in Fig. 2. No substantial differences have been observed for short times; for long times, however, the influence of the external concentration is detectable.

Analogous kinetic behaviour has been observed for grafted EPDM (Fig. 3); in this case, films of different thicknesses (30, 70, 120 and 180 μm) could be tested, but that could not be done with EPDM. As a matter of fact, while it is possible to obtain films from both materials, at low crosslinking degrees only the grafted (and in particular the thin film) samples maintain such a mechanical consistency that they may be handled without damage and without undergoing material loss. The reason is that, in addition to the crosslinking chemical bonds between the elastomeric chains of the grafted polymer, physical bonds exist because of hydrogen bonds between the carboxyls [4].

This is also proved by the kinetic behaviour of q_m vs time shown in Fig. 4 for a grafted sample in comparison with a non-grafted one. By extrapolating q_m at time zero, this tends to ∞ for EPDM (the non-crosslinked sample dissolves), whereas it tends to a finite value for the grafted sample owing to the permanent bonds that exist, because of grafting, between the rubber chains. Under the same time conditions, factor q_m of the two samples does not help in getting further information from the data of Fig. 4, the thickness and the surface being different. The behaviour of q_m vs time may be also different owing to the presence of acid functions that accelerate crosslinking [18, 20, 21].

Figure 5 shows the experimental results arranged for EPDM according to Eqn. (11). It may be concluded that, at least for concentrations of 2 and 4 g S_2Cl_2/l, the kinetic scheme (which excludes diffusion as a limiting process) is not adequate.

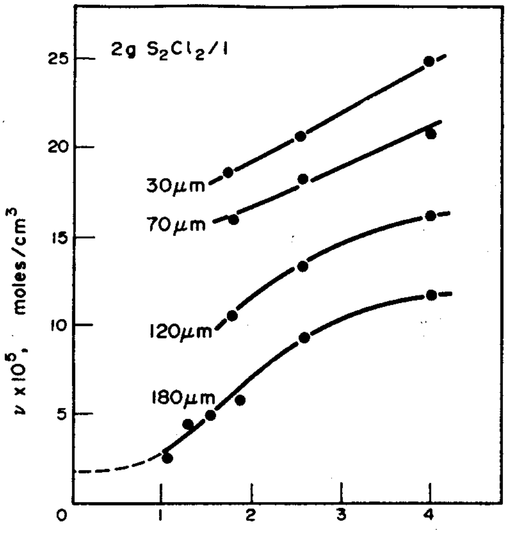


Fig. 3. Density of the elastically effective chains (ν) vs time for grafted EPDM films with different thicknesses. S_2Cl_2 concentration 2 g/. S_2Cl_2/double bonds molar ratio = 10.

Also the Hill Hermans mechanism [22, 23], which is entirely governed by diffusion and which supposes that all active centres react where the reagent penetrates, is inadequate. In fact, this mechanism considers a sharp reaction line that shifts with time. If time is the same and test-pieces have different thicknesses, the reaction line for them all should always have an equal coordinate and ν = Const. should be obtained in the case

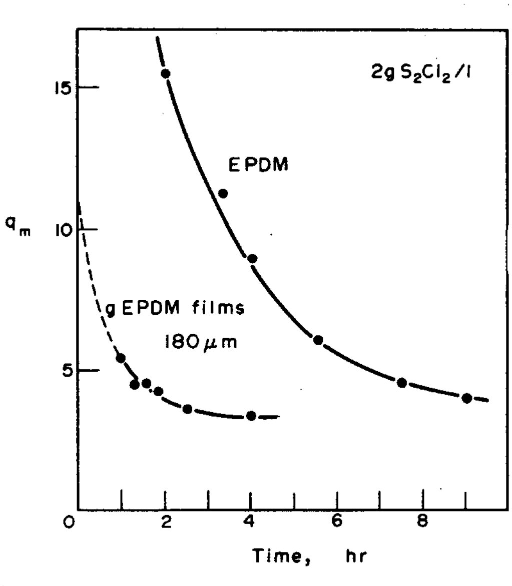


Fig. 4. Degree of swelling q_m vs time for the grafted EPDM films, 180 μm thick, of Fig. 3, and crosslinked EPDM with S_2Cl_2 conc.: 2 g/l of Fig. 1.

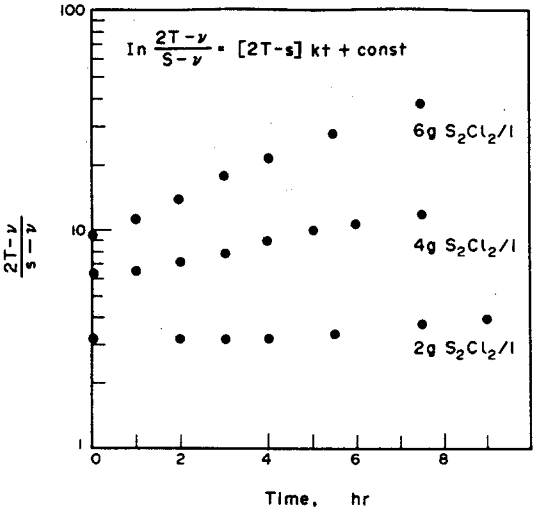


Fig. 5. Kinetics of first order with respect to the double bonds and to S_2Cl_2 reagent for EPDM.

of EPDM; actually, the polymer portion that has not been reached by S_2Cl_2 should dissolve or, in the case of grafted EPDM, it should swell abnormally. Instead, time being the same, ν_{mean} is in grafted films an inverse function of thickness (Fig. 3). Furthermore, our data do not indicate linearity between ν and $\sqrt{C_{\text{ext}}}$, as would be expected by the quoted theory. However, other mechanisms limited by diffusion are possible.

The order of magnitude of the diffusion coefficient calculated by the Fick equation at a time that corresponds to the beginning of steadiness, when the external concentration is not yet markedly changed, may be compared to that obtained by the Scheibel [24] and Le Bas [25] equations for S_2Cl_2 in n-butylacetate, which is $2{\cdot}5 \times 10^{-5}$ cm²/sec.

Table 2 reports the values of D calculated from the experimental curves of Figs. 1, 2 and 3, assuming validity of the Fick law and supposing the diffusion is going on in EPDM from the two main surfaces towards the inside; in the case of grafted films, which are adhering to the slide, it is supposed that the diffusion is from one single surface. It is known [26] that the diffusion coefficients of a solute in a solvent-swollen polymer are 5–10 times lower than the diffusion coefficients of the solute in the pure solvent.

Table 2. Values of the diffusion coefficient of S_2Cl_2 in EPDM and in grafted EPDM, swollen in n-butylacetate

Type of test		D (cm²/sec)
Curve 1 of Fig. 1	2 g S_2Cl_2/l	$1{\cdot}3 \times 10^{-6}$
moles S_2Cl_2/double bonds	6·28 : 1	
Curve 2 of Fig. 1	4 g S_2Cl_2/l	$1{\cdot}3 \times 10^{-6}$
(curve 2 of Fig. 2)		
moles S_2Cl_2/double bonds	6·28 : 1	
Curve 3 of Fig. 1	6 g S_2Cl_2/l	$1{\cdot}6 \times 10^{-6}$
moles S_2Cl_2/double bonds	6·28 : 1	
Curve 1 of Fig. 2	4 g S_2Cl/l	$1{\cdot}2 \times 10^{-6}$
moles S_2Cl_2/double bonds	3·14 : 1	
Curve 3 of Fig. 2	4 g S_2Cl_2/l	$1{\cdot}4 \times 10^{-6}$
moles S_2Cl_2/double bonds	12·56 : 1	
Film, 180 μm, of Fig. 3	2 g S_2Cl_2/l	$0{\cdot}95 \times 10^{-6}$
moles S_2Cl_2/double bonds	10 : 1	

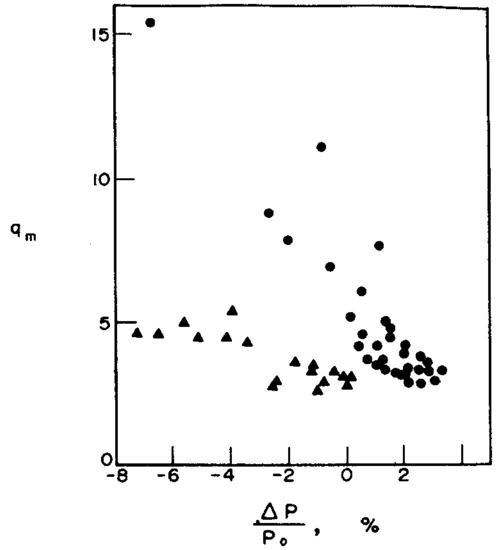


Fig. 6. Variation wt.% of EPDM and grafted EPDM for different degrees of swelling q_m. ● EPDM; ▲ g EPDM films.

It thus appears certain that diffusion plays a very important role in the crosslinking reaction.

During swelling in benzene, both EPDM and grafted EPDM may undergo a weight decrease, due to extraction of non-crosslinked rubber chains; on the other hand, during crosslinking only a weight increase takes place. Figure 6 shows the wt. % variations in respect of the initial weight ($\Delta P/P_0$ %) vs q_m. While for EPDM the value of $\Delta P/P_0$ % reported is that at equilibrium, for grafted EPDM, $\Delta P/P_0$ % is that reached after 24 hr. Two distinct regions may be detected in the figure for the two materials, i.e. q_m being the same, $\Delta P/P_0$ % is higher in grafted EPDM. The reason may be that grafting by transfer may cause rupture of the elastomeric chains.

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Résumé—Le caoutchouc EPDM gonflé d'acétate de n-butyle, est réticulé avec une solution de S_2Cl_2 dans l'acétate de n-butyle. On calcule à partir des mesures de gonflement dans le benzène et l'équation de Flory–Rehner, la densité des chaînes élastiquement actives dans le réseau, en fonction du temps. La cinétique est discutée; le processus de diffusion de S_2Cl_2 dans le polymère gonflé joue un rôle très important. On a réticulé l'EPDM greffé de poly(acide acrylique) sous forme de films minces en utilisant le S_2Cl_2 comme réactif et l'acétate de n-butyle comme agent gonflant. On a observé le même comportement cinétique. Les liaisons intermoléculaires sont dues à la fois aux ponts de soufre dans l'EPDM et aux ponts de soufre et d'hydrogène dans les échantillons d'EPDM greffés.

Sommario—La gomma EPDM, rigonfiata in n-butilacetato è stata reticolata con soluzioni di S_2Cl_2 in n-butilacetato. La densità delle catene elasticamente efficaci è stata valutata in funzione del tempo da misure di rigonfiamento in benzene. Viene discusso lo schema cinetico: il processo di diffusione di S_2Cl_2 nel polimero rigonfiato ha un ruolo molto importante. La reticolazione di EPDM innestato con poliacido acrilico è stata effettuata su film sottili usando come reagente S_2Cl_2 e n-butilacetato come rigonfiante mostrando un comportamento cinetico simile. I legami intermolecolari risultano dovuti nell'EPDM a ponti di zolfo e nell'EPDM innestato a ponti di zolfo e a legami idrogeno.

Zusammenfassung—In n-Butylacetat gequollenor EPDM-Gummi wurde mit einer Lösung von S_2Cl_2 in n-Butylacetat vernetzt. Aus Quellungsmessungen in Benzol und unter Anwendung der Gleichung von Flory–Rehner konnte die Dichte der Ketten des Netzwerkes, die zur Elastizität beitragen, als Funktion der Zeit berechnet werden. Die Kinetik wird diskutiert; die Diffusion des S_2Cl_2 im gequollenen Polymeren spielt eine bedeutende Rolle. Vernetzung von EPDM, gepfropft mit Polyacrylsäure, wurden mit S_2Cl_2 an dünnen Filmen mit n-Butylacetat als Quellungsmittel vorgenommen; sie zeigen das gleiche kinetische Verhalten. Intermolekulare Bindungen bestehen sowohl aus Schwefelbrücken in EPDM wie aus Schwefelbrücken und Wasserstoffbrücken in den gepfropften Proben von EPDM.