NOTES

Study of Thermal Properties and Morphology of Interpenetrating Polymer Networks from Natural Rubber and Polyacrylamide

The historical synthesis of the interpenetrating polymer network (IPN) by Millar¹ and the pioneering studies of Frisch et al.² and Sperling and Widmaier³ have established the genuine interlocking of two or more cross-linked polymer chains. Hourston and Romaine⁴ reported a series of IPNs from natural rubber and styrene with *p*-divinylbenzene as the cross-linker. In the present study, we report the thermal properties and morphology of a series of IPNs from natural rubber and polyacrylamide with ethylene glycol dimethylacrylate as the cross-linker.

EXPERIMENTAL

Materials

The materials used are summarized in Table I.

Polymerization

The recipe used in the study is shown in Table II.

Latex was stabilized with ammonia, and acrylamide, sodium lauryl sulfate, and ethylene glycol dimethacrylate was added to the latex with stirring. When complete dispersion was effected, ammonium persulfate was stirred in and the reaction vessel was closed and kept undisturbed for 24 h. The reacted latex was then coagulated using 10%

Journal of Applied Polymer Science, Vol. 49, 2229–2233 (1993) © 1993 John Wiley & Sons, Inc. CCC 0021-8995/93/122229-05 formic acid solution. The coagulum was filtered, washed, dried, and weighed.

Techniques

- (a) Sol/gel ratio determination: Soxhlet extractions were performed on pieces (ca 0.5 g) of the dried sample to extract linear and grafted material. A two-solvent process involving, first, petroleum ether, which was used to extract natural rubber and natural rubber-rich material, and then water, used to extract polyacrylamide and polyacrylamide-rich material.
- (b) DSC experiments were performed on a Perkin-Elmer DSC-7 at a heating rate of 10°C min⁻¹.
- (c) Studies on the morphology of the IPNs were carried out using a Philips scanning electron microscope (Model XL-30). For these studies, samples of the IPNs were fractured in liquid nitrogen and vacuumcoated using gold-palladium plasma for a period of 2 min.

RESULTS AND DISCUSSION

The thermal behavior of IPNs was examined through DSC curves (Fig. 1). In the case of (a) natural rubber, the maximum decomposition temperature was 374.20° C. However, the IPN-b and IPN-c showed the maximum decomposition temperature at 374.40 and 375.40° C, respectively. From Figure 1 it is observed that the IPN-d with a high acrylamide content exhibited three endothermic peaks at 283° C (peak I), 374.20° C (peak II), and 475.40° C

| Table I Materials Used in This Study | Tab | ole I | Materials | Used in | This Study |
|--------------------------------------|-----|-------|-----------|---------|------------|
|--------------------------------------|-----|-------|-----------|---------|------------|

| Materials | Description | Source | Code |
|--------------------------------|--------------------------|--|---------------|
| Latex (natural rubber) | Dried rubber content 29% | Rubber Research Institute of Malaysia | NR |
| Acrylamide | | BDH | AC |
| Ethylene glycol dimethacrylate | Acrylic cross-linker | BDH | EDM |
| Ammonium persulfate | Initiator | BDH | AP |
| Sodium lauryl sulfate | Emulsifier | Merck | \mathbf{LS} |
| Formic Acid | Chemical coagulator | BDH | FA |

| | Parts by Weight | | | |
|-----------------------|-----------------|-------|-------|--|
| Ingredients | IPN-b | IPN-c | IPN-d | |
| Latex (29% DRC) | 34.75 | 24.75 | 14.75 | |
| Acrylamide | 15.00 | 25.00 | 35.00 | |
| Sodium lauryl sulfate | 0.50 | 0.50 | 0.50 | |
| Ethylene glycol | | | | |
| dimethyacrylate | 0.25 | 0.25 | 0.25 | |
| Ammonium persulfate | 0.50 | 0.50 | 0.50 | |

Table II Recipe Used in This Study

Temperature: room temperature (29°C). Period of reaction: 24 h.

(peak III). Frisch et al.⁵ studied the thermal behavior of various IPNs and found that they possessed greater thermal stability than that of their individual component net-

| Table III | Data | Arising | from | the | Two-so | lvent |
|------------|------|---------|------|-----|--------|-------|
| Extraction | Proc | edure | | | | |

| | Weight Extracted (%) | | | | |
|--------|----------------------|-------|-------|--|--|
| Sample | Petroleum Ether | Water | Total | | |
| IPN-b | 65.8 | 10.8 | 76.6 | | |
| IPN-c | 57.1 | 13.8 | 70.9 | | |
| IPN-d | 0.0 | 31.1 | 31.1 | | |

works. In the present study, the thermal behavior of IPNb to IPN-d suggests that the IPNs possess greater thermal stability than that of natural rubber.

Scanning electron micrographs at 386 magnification for natural rubber (at 385 magnification) and IPN-b-IPNd (at 386 magnification) are shown in Figures 2 and 3.



Figure 1 DSC curves of (a) NR; (b) IPN-b; (c) IPN-c; (d) IPN-d.



(a)



Figure 2 SEM micrographs of (a) NR; (b) IPN-b.

(b)



(c)





These IPNs show two distinct domains of an individual component network, indicating the phase separation.

To determine the sol/gel ratio, a two-solvent Soxhlet extraction procedure, initially with petroleum ether and, finally with water, was used. See Table III for the data derived from this extraction procedure. For the IPN-b material, the petroleum ether extraction removes 65.8% by weight of the test sample. For the IPN-d sample, the total extraction removes 31.1% by weight of the test sample.

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