

Mastic of Polymer-Modified Bitumen and Poly(vinyl chloride) Wastes

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ABSTRACT: Micronized poly(vinyl chloride) (PVC) pipe waste has been employed as a soft filler in making bituminous products. The binder used in this study consists of styrene-butadiene-styrene-modified bitumen, isocyanate production waste, and an antioxidant. The time-dependent properties of these binders were assessed by constructing a master curve using frequency multiplexing. A plateau at intermediate and low frequencies in the storage modulus curves of modified bitumen implies the existence of molecular association among the constituents compared to neat bitumen. The enhanced adhesion between the PVC particles activated by H_2O_2 and the bitumen was verified in terms of bitumen anchorage onto particles after washing in hot tolu-

ene. Confirmation of these effects can also be seen by increased stiffness of the bituminous mix with the activated particles over that with nonactivated particles. Consequently, a suitable mastic composition consisting of these activated coarse and micronized particles, limestone dust, and polymeric bituminous binder has been achieved. The results indicate that the optimized mix can be used as waterproof roof mastic as an alternative to conventional bituminous mastic. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 90: 1347–1356, 2003

Key words: polymeric bitumen; PVC waste; mastic; frequency multiplexing; waterproofing

INTRODUCTION

Bitumen has been known to be used as waterproofing materials in buildings for a long time. However, the performance of these bituminous systems is questioned, given that they are brittle and hard in cold environments and soft in hot environments.¹ It has been recognized that these deficiencies of bitumen can be overcome by the addition of polymers for improving viscoelastic behavior besides maintaining its own advantages.^{2–5} However, the high cost of polymers has been an inhibitory factor for mass-scale adoption in place of conventional bitumen even though their use is profitable in terms of cost–benefit ratio.⁶ The improved low-temperature ductility/resiliency and retention of molecular associations (more viscous) at high temperatures of bitumen can also be achieved by the addition of plastics waste in micronized form.^{7,8} The activation of this finely ground plastics waste either by copolymer grafting or ozonization techniques makes them reactive toward bitumen. This has resulted in good compatibility with bitumen by reducing particle size and ensuring their anchorage. With

the increased usage of plastics in various sectors, the discharge of plastics wastes in huge quantities has serious implications on society from an environmental perspective. Therefore, adding suitably sized activated plastics waste to bitumen formulation opens new prospects in this field to be used as modifier/fine aggregate, thus disposing of the waste while making value-added products.

In the present study, poly(vinyl chloride) (PVC) pipe waste was used as a filler for making bituminous roof mastic up to a level of 60–70 wt %. The main reason for its selection is that after secondary recycling, the impairable properties of PVC could not economically be achieved even by adding increased doses of additives, thus becoming a burden to the environment. To achieve gainful use, the approach of bitumen grafting onto PVC waste particles was chosen (after their activation) through introducing oxygenated compounds into structures. Previously, chemical anchoring of polybutadiene/ethylene-propylene-diene monomer onto recycled polyethylene was reported to stabilize the polyethylene-modified bitumen suspension.⁹ Ozonization of polyethylene waste has also been employed to improve its adhesion with bitumen. However, the reported addition of activated waste particles into bitumen was up to only about 10 wt %.¹⁰ In the absence of adequate compatibility, the modified bitumen loses its ultimate properties and the existence of the separated phase induces high crack susceptibility. A survey on a flat roof¹¹ indicates that mastic asphalt is more durable as a roof covering than the

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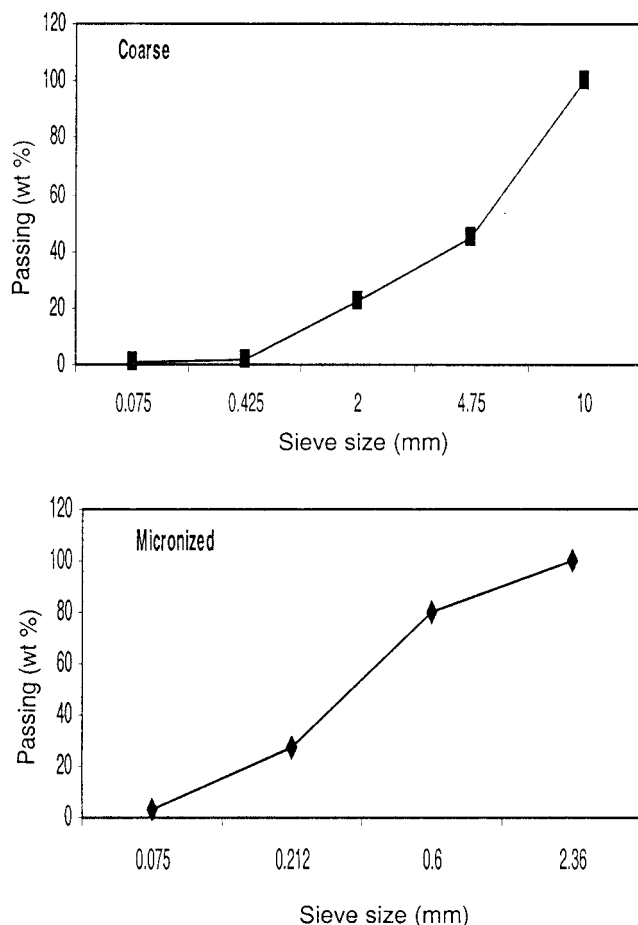


Figure 1 Sieve analysis of PVC pipe waste particles.

built-up bitumen felts. However, some evidences of failure were observed when the mastic was subjected to varying climatic conditions with respect to rain penetration. To meet the basic necessary requirements of mastic such as water impermeability, flexibility, and adequate workability, bitumen modified with rubber/activated plastics waste can be used in place of standard bitumen.^{12,13}

In this study, we report the use of activated micronized PVC pipe waste on the properties of polymer-modified bitumen. The rheology of modified binders is examined under frequency multiplexing. The physico-mechanical and waterproofing properties of bitumen-PVC waste mastic are also discussed in relation to existing standard specifications.

EXPERIMENTAL

Materials

Shredded (coarse) and micronized (fine) PVC pipe waste materials were obtained from the recycling plant. The sieve analysis of these waste particles is shown in Figure 1. The average particle sizes of coarse and micronized waste were 4.8 and 0.37 mm, respec-

tively. The crushing strength was 2.4% (Table I). An industrial grade bitumen, 85/25, was procured from an Indian supplier (softening point, 82°C; penetration at 25°C, 16 dmm; ductility at 27°C, 3 cm; flash point, 200°C; loss on heating, 0.30%). Isocyanate production waste was received from an Indian manufacturer of toluene diisocyanate. Chemically, it contained about 98% carbon, traces of isocyanates, and other organic matters. The average particle size of ground waste was 0.51 mm. Styrene-butadiene-styrene (SBS) was procured from M/s Shell Chemical Ltd.

Preparation of samples

The polymer-modified bitumen was prepared by adding SBS elastomer (3 wt %) into bitumen under an optimized condition of blending time (60 min) and temperature (180°C).¹⁴ Subsequently, the isocyanate production waste was compounded with modified bitumen and stirred for 2 h in a blender at a speed of about 2000 rpm with three blades of 20-mm diameter. Before inclusion, the waste particles were treated with stearic acid (1 wt % of particles) to obtain their homogeneous dispersion in a bitumen matrix. The resultant blend was divided into several containers and stored at room temperature for further work.

The coarse and fine PVC waste particles were thoroughly washed and dried in an oven at 60°C to remove the foreign matter. The finely ground PVC particles were suspended in H₂O₂ solution (50% w/v). The peroxidation reaction was carried out in a reaction vessel at 50°C for 30, 60, and 120 min under continuous stirring. The suspension was then filtered after a specified time interval. The treated particles were dried at room temperature. The efficacy of the activation was examined in terms of anchored bitumen onto particles.

Preparation of mastics

The bitumen-elastomer mastic consisted of SBS-modified bituminous binder (20–30 wt %) activated PVC waste particles (~ 60–70 wt %), limestone dust

TABLE I
Properties of PVC Waste Particles

Property	Value
Density (g/cm ³)	
Coarse	0.88
Micronized	0.72
Water absorption % (24 h)	Nil
Average particle size (mm)	
Coarse	4.80
Micronized	0.37
Crushing strength % (IS: 2386-90 Part II)	2.4
Impact value (IS: 2386-90 Part IV)	No breakage
Soundness (IS: 2386-90 Part V)	Not disintegrated

TABLE II
Composition of Bitumen-PVC Waste Mixes

Sample	Composition (wt %)					
	Polymeric bitumen	Coarse PVC	Micronized PVC	Isocyanate waste	Limestone dust	Antioxidant
1	30	70	—	—	—	—
2	30	35	35	—	—	—
3	30	35	28	—	7	—
4	30	35	20	—	15	—
5	30	—	70	—	—	—
6	30	—	63	—	7	—
7	30	—	55	—	15	—
8	23	28	28	16	4	1

(~ 7–15%), and antioxidant additives (~ 1–2 wt %). The tentative compositions of mastic are given in Table II. The polymer-modified bitumen was heated at 180°C. Thereafter, antioxidant (resorcinol and hydroquinone) was added under constant stirring. Subsequent to this, inclusion of limestone dust was made and mixed thoroughly for 60 min followed by addition of coarse and fine PVC particles. The prepared mix was then cast into the desired size of molds under adequate tamping. The demolding of samples was done after 72 h for the purpose of testing.

Methods

The linear viscoelastic response of the modified bituminous binders was evaluated on a stress-controlled rheometer (AR 1000, TA Instruments, New Castle, DE) using 20-mm parallel-plate geometry with 2-mm gap. The measurements were taken from 20 to 100°C temperatures with an increment of 10°C at frequencies ranging from 0.01 to 100 Hz with three points per decade. The strain of 0.05 was selected to remain in the linear viscoelastic region over the entire temperature and frequency range.

A dynamic mechanical analyzer (DMA 2980; TA Instruments) was used to examine the effect of activation on the viscoelastic properties of the bituminous mix. The dual-cantilever beams (size: 20 × 14 × 5 mm) were tested in the temperature range of 35 to 100°C at a heating rate of 2°C/min and 5- μ m amplitude. The storage modulus and $\tan \delta$ of the samples were recorded.

The diffuse reflectance spectra of the control and the activated PVC particles were recorded on a UV-visible spectrophotometer (UV 246, Shimadzu, Kyoto, Japan). Surface morphology of the activated and the bitumen-grafted particles was examined by a scanning electron microscope (Leo 435 VP). Before examination under microscope, the particles were coated with gold/palladium to render them conductive.

The hardness number of mastic was measured according to IS 1195-90.¹⁵ A load of 100 kg/cm² was

applied on a sample of 25 mm thickness and 100 mm diameter for 1 min through a flat-ended indentation pin (6.35 mm diameter). The depth of indentation was recorded at temperatures of 25 and 45°C. The flexural properties of mastic were determined according to ASTM D 790-86 on an Instron testing machine (Model 1342). The load-deflection curve was recorded at a crosshead speed of 5 mm/min under a span length of 200 mm. The compressive test of mastic was performed on a hydraulic machine. The average value of five specimens was taken for all the tests.

Waterproofing properties of mastic were assessed in terms of water permeability, water absorption, and rain penetration. The water permeability test on mastic was conducted according to IS 5913-1995.¹⁶ The lowering of the water meniscus in 30-cm-long glass tubes of 2.5 cm diameter, fixed at the center of the flat mastic block, was noted after 7 days. The rain penetration test on mastic was carried out under simulated rain conditions. The seepage of water on the rear side of the mastic was observed after 15 and 30 min. The water absorption of mastic was also tested after immersion in water for 24 h.

The thermal performance of mastic (30 × 30 × 2.5 cm) was measured in terms of thermal conductivity using a guarded hot-plate conductivity apparatus according to IS 3346-90.¹⁷

RESULTS AND DISCUSSION

Bituminous binder

The frequency dependency of bituminous binder is shown in Figure 2. It was found that storage modulus tends to slope upward with increasing frequency. As the temperature increases, the binder becomes softer and goes through transitions that increase the available space for molecular motions. Consequently, the increased slope in the plots at intermediate frequencies is observed. It is noted that the loss modulus decreases at lower temperatures (20–40°C) and increases at higher temperatures as the frequency in-

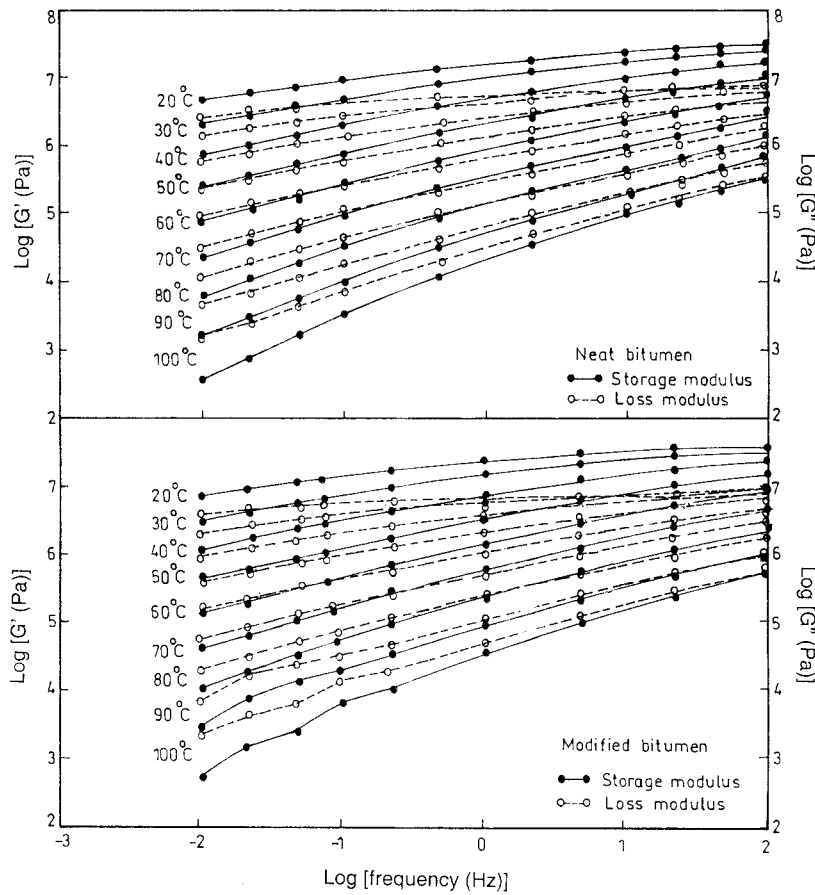


Figure 2 Frequency sweep of neat and modified bitumen as a function of temperature.

creases. At higher temperatures, the ratio of loss modulus to storage modulus is more at low frequencies than the ratio at higher frequencies. Using the time-temperature superposition principle, a shift factor was calculated from the frequency multiplexing plot by

shifting the viscoelastic data with respect to time for each temperature to construct a master curve. Figure 3 shows the storage modulus master curves of bituminous binders at a reference temperature of 50°C. It is clearly seen that the storage moduli of the modified

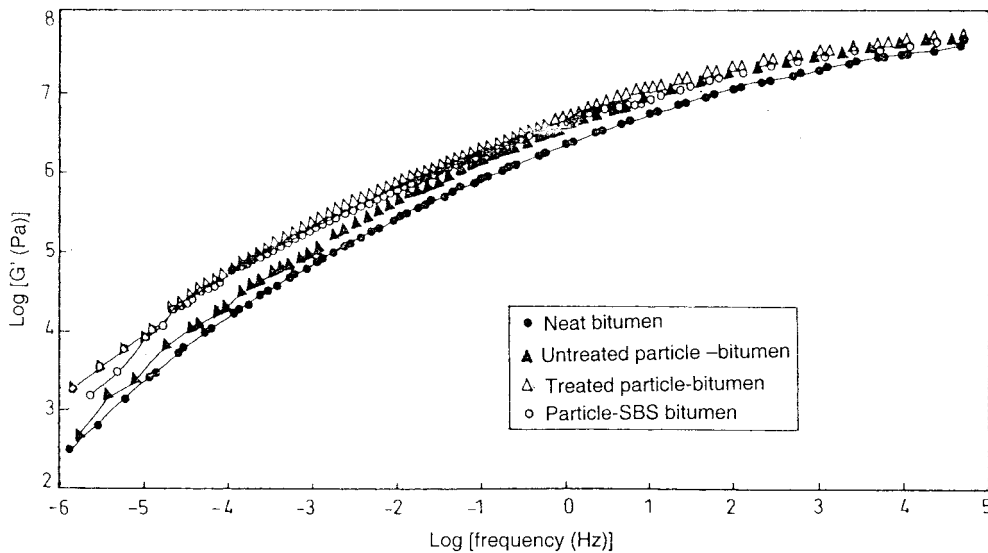


Figure 3 Storage modulus master curves for neat and modified bitumen at a reference temperature of 50°C.

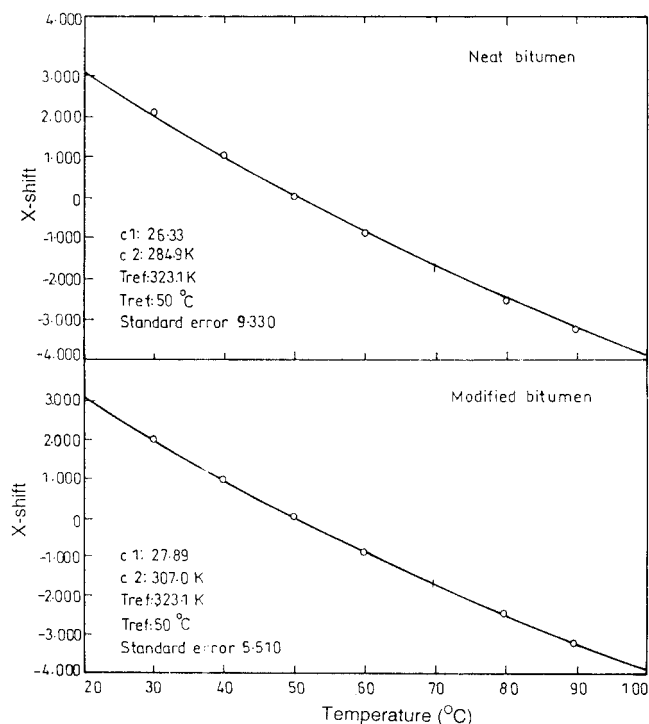


Figure 4 Plots of shift factor of neat and modified bitumen as a function of temperature.

bitumen have higher values than those of the unmodified bitumen over the entire range of frequency. The more effective changes in the neat and modified bitumen are observed at low and intermediate frequencies, probably attributable to the modification effects. At higher frequencies, the storage moduli of all the modified binders are almost the same ($\sim 10^7$ Pa). In the intermediate frequencies, the modified binders present a plateau region that extend the apparent shift of terminal zone to the lower frequencies. This fact could be attributed to the possible formation of molecular entanglements among the macromolecular components as reported previously for polymer-modified bitumen.^{18,19}

The thermal susceptibility of bituminous binders was also assessed by plotting a curve between the shift factor and the temperature using the Williams-Landel-Ferry equation²⁰ (Fig. 4). The values of universal constants C_1 and C_2 were 26.33 and 284.90 K for the neat bitumen and 27.89 and 307 K for the modified bitumen, respectively. It is observed that there is an acceptable resemblance between theoretical and measured shifts at all temperatures. It is noted that the slope in the curve of the modified bitumen is slightly less than that of the neat bitumen, thus showing superior temperature resistance behavior. Similar results were reported previously on the modified bitumen at temperatures well above the glass transition.²¹

Optimization of mix

The optimization of coarse and micronized PVC wastes in bituminous binders was carried out on the basis of hardness number (Table III). Bituminous mix prepared from coarse particles showed a hardness number at a scale of 13 at 25°C and 40.80 at 45°C, respectively. The improvement in hardness number of mixes was observed when the binder was mixed with a combination of coarse and micronized particles along with limestone dust filler as a minor constituent. The hardness number of the resultant mix was reduced from 13 to 8 at 25°C with respect to coarse particles-bitumen system as a result of filling of gaps between the larger particles. Increasing the percentage of fine filler reduced the hardness number of mixes. Bituminous mix composition containing only micronized PVC particles showed a hardness number of 9.80 at 25°C. The slight reduction in this value was observed on further addition of limestone dust (~ 15 wt %). On the contrary, rigidity of the mix improved significantly with the addition of limestone dust at 45°C. This is attributed mainly to the homogeneous synergistic presence of filler/fine mineral in the bitumen medium. The large-size particles dispersed irregularly in the bitumen possessing only a localized ri-

TABLE III
Optimization of Bitumen-PVC Waste Mixes

No.	Sample	Hardness number	
		25°C	45°C
A. Bitumen-coarse/micronized particles mix			
1	Bitumen + coarse particles	13	40.80
2	Bitumen + coarse and micronized particles + mineral filler (7%)	9.20	27.60
3	Bitumen + coarse and micronized particles + mineral filler (15%)	8	20.80
B. Bitumen-micronized particles mix			
4	Bitumen + micronized particles	9.80	20.90
5	Bitumen + micronized particles + mineral filler (7%)	11.40	17.60
6	Bitumen + micronized particles + mineral filler (15%)	8.90	15.20

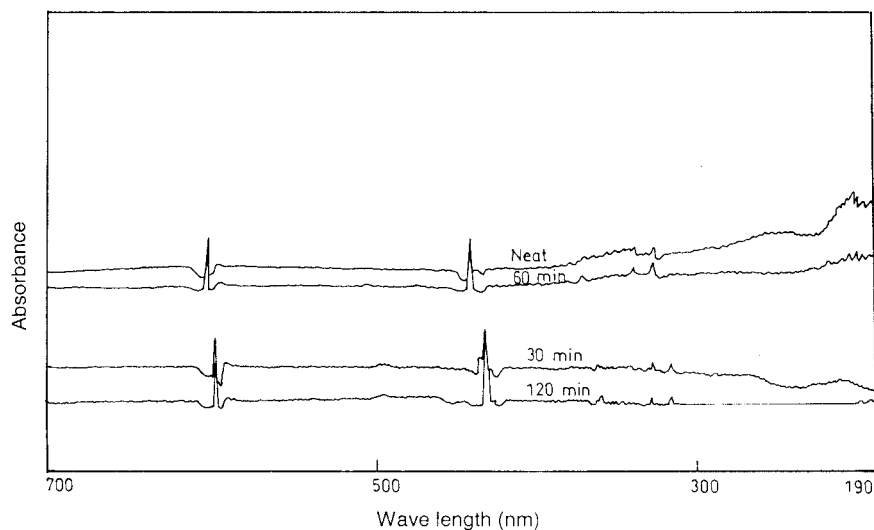


Figure 5 Diffuse-reflectance UV-visible spectra of neat and activated PVC particles.

gidity to the system and were responsible for varying hardness numbers at different locations of the mixes. It was found that bituminous mix containing coarse and micronized particles gives satisfactory results.

Effect of activation

The diffuse-reflectance UV-visible spectra of control and activated PVC particles are shown in Figure 5. The magnitude of absorbance near 220 nm decreases with the increase of treatment time. After a 2-h treatment, there was a complete disappearance of the absorption band, probably attributable to breaking of the C—Cl bond and vinyl sequences. This indicates that peroxidation reaction leads to a decrease in the molecular mass of PVC after the peroxide radical attacks. The effectiveness of H_2O_2 treatment was further confirmed in terms of particle topography as observed in SEM (Fig. 6). The activated particles seem to be rough and porous in relation to nonactivated ones. The cleavage in the activated particles was very distinct when the peroxidation reaction was extended for 2 h. To ascertain the coupling between the bitumen and the activated particles, the isolated particles from the blend were thoroughly washed with carbon tetrachloride followed by their crystallization in hot toluene to remove the complete physisorbed bituminous fraction. SEM analysis of these washed particles confirmed the entrapment of bitumen ingredients in the activated spongy particles even after severe washing (Fig. 7). The existence of bitumen inside the activated particles can be easily differentiated from the control sample in terms of a continuous dark blackish matrix and the presence of voids on particle surfaces. The control particles seem to be hard contrary to the soft, spongy, slightly brownish, and irregular surface of activated particles. It is noted that the effective anchoring of bitumen could be obtained only after the 2-h

treatment. This behavior could be explained on the basis of either a deactivation of particles by bitumen (grafting) or a free-radical coupling reaction. It is

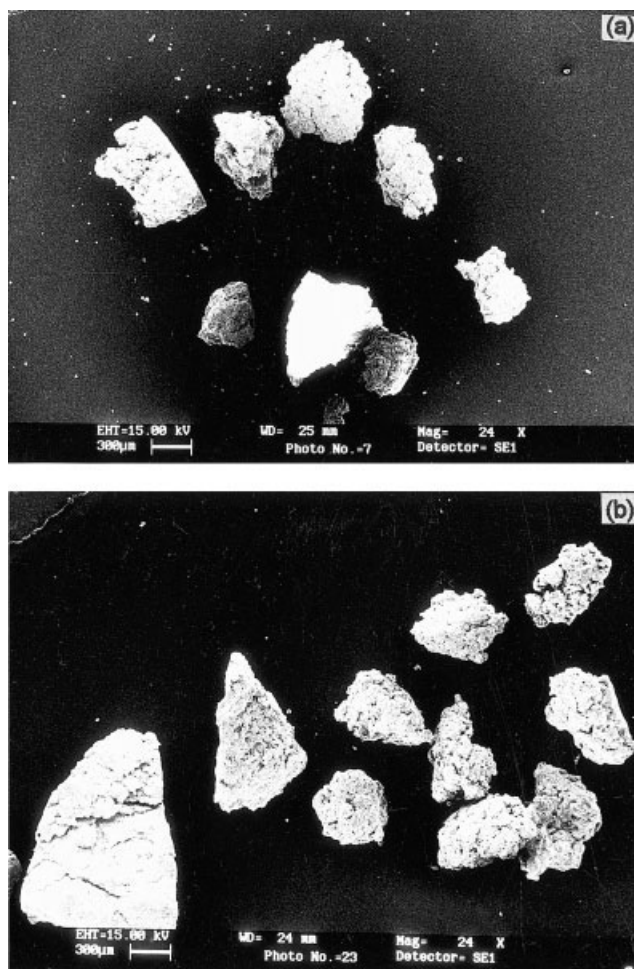


Figure 6 SEM of PVC waste particles: (a) control, (b) activated (2-h treatment).

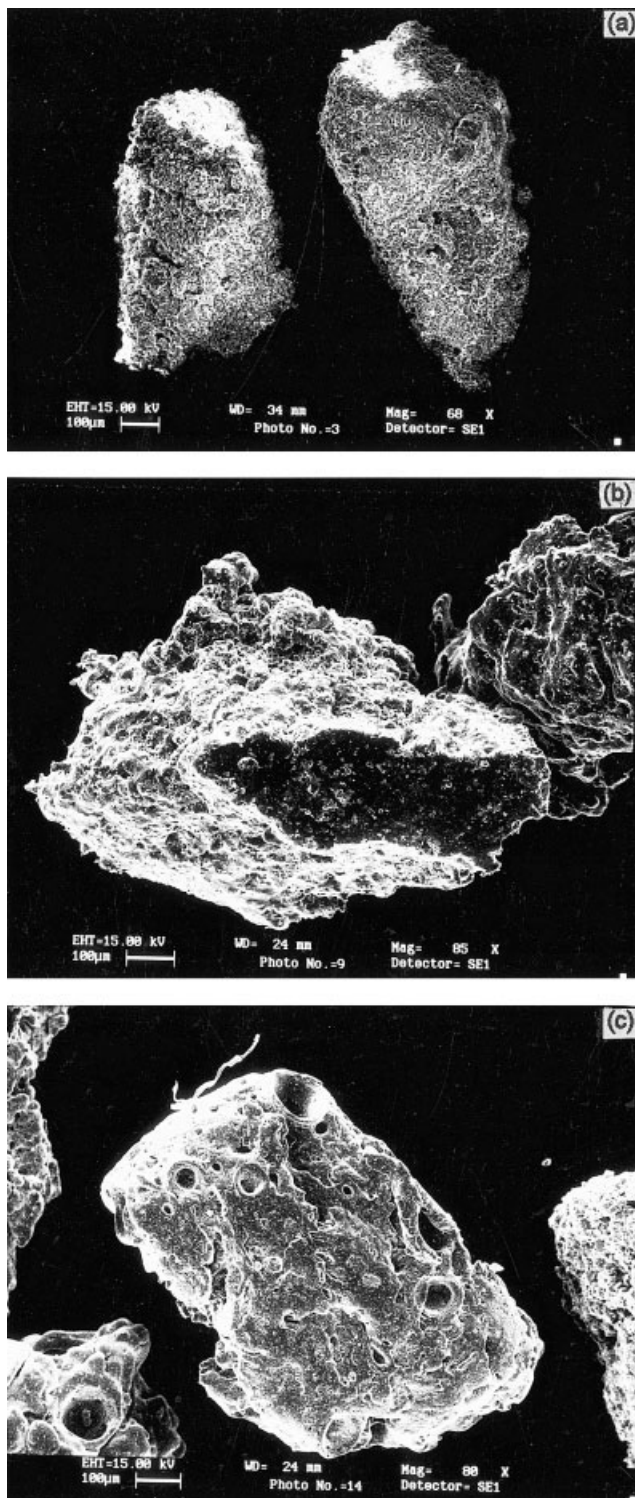


Figure 7 SEM of grafted bitumen onto PVC particles after washing: (a) control, (b) activated (1 h), (c) activated (2 h).

known that reaction of plastics with oxidizing chemicals would produce reactive chemical species on the surface of plastics, which could participate in the subsequent reaction medium.²² The enhanced anchoring of bitumen onto particles is probably attributable to the presence of increased coverage of O⁻ and COO⁻

TABLE IV
Performance of the Activated PVC Waste in Bituminous Binders

System	Hardness number	
	25°C	45°C
A. Polymer-modified bitumen		
• Micronized particles		
Nonactivated	5.40	22.27
Activated	3.25	9.50
• Coarse and micronized particles		
Nonactivated	9.17	33.77
Activated	6.60	29.83
B. Isocyanate waste-filled polymer-modified bitumen		
• Coarse and micronized particles		
Nonactivated	10	40
Activated	8.60	31

sites on the surface of polymers. These radicals have far more chances to deactivate on bitumen. This helps to obtain better compatibility of PVC particles with bitumen to improve the blend's performance.

The performance of the activated particles in bituminous binders was evaluated in terms of hardness number (Table IV). It is observed that the hardness number of a mix containing activated particles improves doubly compared to the mix obtained with nonactivated particles (Fig. 8). The compatibility between the activated particles and the bitumen, which occurred as the result of grafting of bitumen onto particles, could be responsible for the improved behavior. It was also observed that the performance of micronized particles in bitumen is superior to that of the combined mix of coarse and micronized particles. An explanation for this behavior could be the enhanced peroxidation reaction at the grain boundary of micronized particles, contrary to the retarded reaction in the grain core of coarse particles arising from limits of gas diffusion. At 45°C, the retention of stiffness of the activated particle-bitumen system is roughly 2

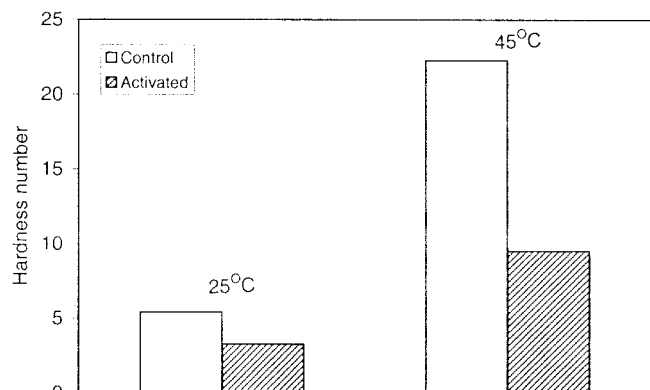


Figure 8 Hardness number of control and activated micronized PVC waste particles containing bituminous mix.

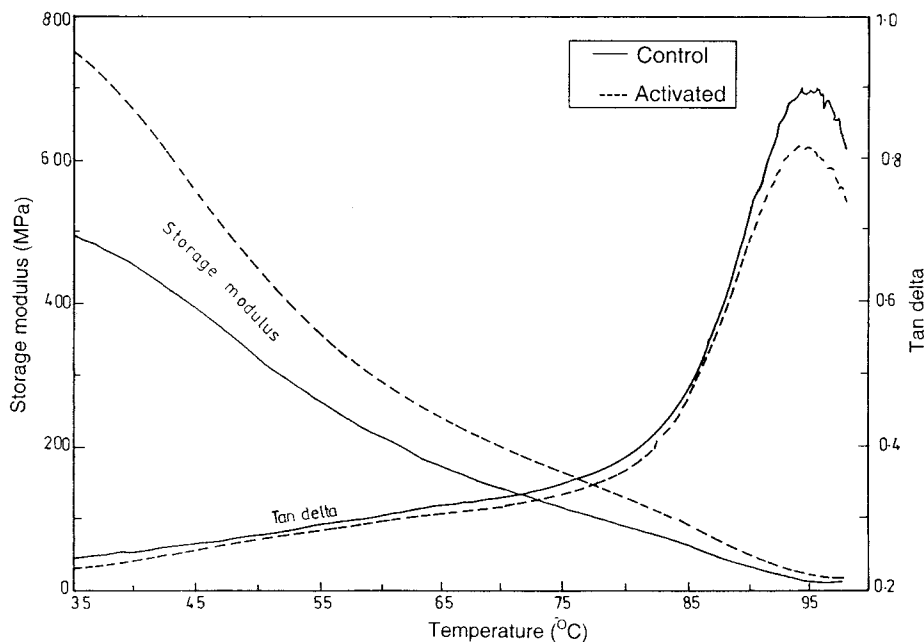


Figure 9 DMA traces of bituminous mastic made from PVC waste particles.

times higher than that of the nonactivated particle mix. On the contrary, the marginal difference in the values of hardness number was observed for mixes containing combined coarse and micronized particles. The results of micronized PVC-bitumen mix are examined in accordance with IS 3037-91.²³ The specified hardness values mentioned in the specifications are 2–8 at 25°C and 10–65 at 45°C. It was found that properties are comparable and meet the hardness requirement. The efficacy of particle treatment in bituminous binder can also be seen in terms of viscoelastic response (Fig. 9). The difference in the values of storage modulus and loss tangent clearly indicates the increased affinity of activated particles toward bitumen. The magnitude of activation is more pronounced at low temperatures than that at higher temperatures. It was observed that the activated PVC-bitumen mix is stiffer and more elastic than the mix containing nonactivated particles. The desired flexibility of mixes attributed to the presence of activated PVC particles may help to avoid the crack developments by thermal expansion/contraction and variation in climatic conditions.

Roof mastic

To meet the satisfactory performance of roofs, the waterproof mastic layers should be stiff enough at elevated service temperatures, possess good stripping resistance, sufficient flexibility at low temperatures, desirable thermal conductivity, and good time-independent properties. In view of unsatisfactory experience using traditionally adopted hardness measurement as a mean of quality assessment, knowledge on

other performance-based properties is also desirable to assess the suitability of materials.

The compressive strength of mastic made from only micronized particles is slightly lower than the mastic obtained with the combined coarse and micronized particles. The flexural strength of a mix of coarse and micronized particles is about 44% higher than the micronized particles mix. However, the flexural modulus of the combined coarse and micronized particles mix is marginally lower than that of the micronized particles mix. This is reflected in the load-deflection curves of mastic (Fig. 10). The deflection in the mastic made from the combined coarse and fine particles is quite appreciable. The energy to break (toughness) of mastic is also improved. The improved stiffness of the micronized particles mix over the combined coarse and micronized particles-based mastic is supported by flexural modulus data.

The bituminous mix composition composed of coarse and micronized PVC waste, antioxidant, isocyanate waste, mineral filler, rosin, and polymer-modified bitumen has been finalized for making waterproof roof mastic. The physicochemical properties of this mastic are given in Table V. The density of mastic is in the range of 1000–1300 kg/m³. The moisture vapor transmission of bituminous binder is about 11.29 g m⁻² 24 h⁻¹. The water absorption of the micronized particles-bituminous mastic is lower than that of the mastic made from the combined coarse and micronized particles. The water permeability result shows no water seepage at the bottom side of 25-mm-thick mastic slab and a negligible lowering of water meniscus in the measuring cylinder even after 7 days' examination. During simulated rain, there was no ev-

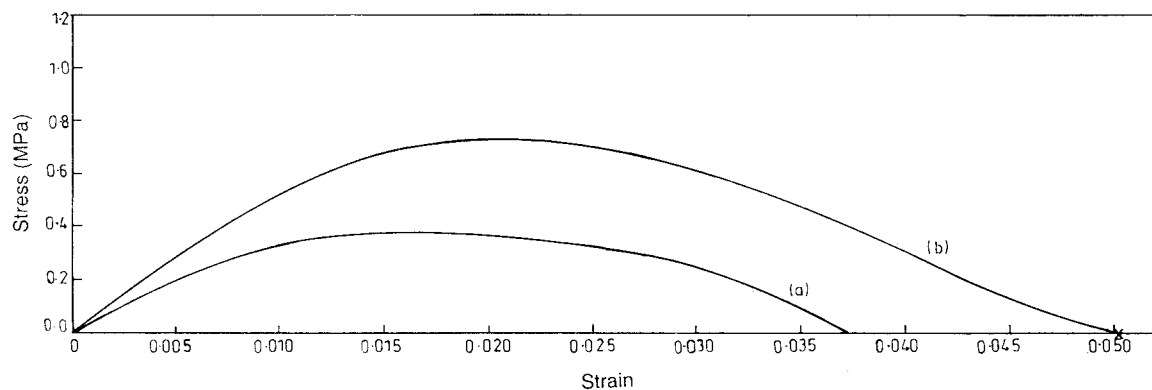


Figure 10 Flexural load-deflection curves of (a) bitumen-micronized PVC particles mix and (b) bitumen-coarse and micronized PVC particles mix.

idence of spalling of particles and/or erosion of binder after a 30-min exposure. The penetration of water on the rear side of mastic slabs was also not observed. Thermal compatibility of mastic with substrates was also improved as a result of reduced coefficient of thermal expansion of the binder compared to that of standard bitumen. The thermal conductivity of PVC waste-bitumen mastic ($0.228 \text{ kcal m}^{-2} \text{ h}^{-1} \text{ }^\circ\text{C}^{-1}$) is significantly lower than the conventionally used materials such as lime concrete ($0.628 \text{ kcal m}^{-2} \text{ h}^{-1} \text{ }^\circ\text{C}^{-1}$) and mud phuska ($0.446 \text{ kcal m}^{-2} \text{ h}^{-1} \text{ }^\circ\text{C}^{-1}$) for roof treatment. This may provide adequate thermal comfort in buildings.

CONCLUSIONS

Rheological studies reveal that the isocyanate waste modified-polymeric bitumen is superior to conven-

tional bitumen. The activation of PVC waste particles by the peroxidation method improves their compatibility with the bituminous binder. Bituminous mastic made from activated particles possesses adequate physicommechanical and waterproofing functions. Thus, the grafting of bitumen onto PVC particles through the activation is essential before inclusion in the bitumen matrix. This contributes toward an alternative route for an effective use of plastics waste as soft filler in bituminous mix for making waterproofing compound in buildings.

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TABLE V
Properties of Waterproof PVC Waste-Bitumen Roof Mastic

Property	Bituminous mastic	
	Coarse and micronized	Micronized
Density (g/cm^3)	1.28	1.30
Porosity (%)	1.52	2.60
Flexural strength (MPa)	6.87	3.87
Flexural modulus (GPa)	0.029	0.033
Energy to break (J)	8.20	3.80
Fracture energy (J/m^2) $\times 10^3$	4.69	2.17
Hardness number		
25°C	6.60	3.25
45°C	29.83	9.50
Compressive strength (MPa)	3.99	2.63
Water absorption (%), 24 h	2.38	1.39
Water permeability (after 7 days)	No seepage	No seepage
Rain penetration (after 30 min)	No spalling of particles	No spalling of particles
Thermal expansion ($^\circ\text{C}$)	1.20×10^{-4}	—
Thermal conductivity ($\text{kcal m}^{-2}\text{h}^{-1}\text{ }^\circ\text{C}^{-1}$)	0.228	—

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