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Use of rerefined oil distillation bottoms as extenders for roading bitumens

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Vacuum distillation bottoms from the rerefining of waste automotive oils (WODB) can be successfully blended with standard petroleum bitumen to produce a stable binder with acceptable physical properties for roading applications. The increase in viscosity and shear susceptibility after accelerated ageing of thin films at 163 °C is slightly less for bitumen extended with WODB than for bitumen itself; but at temperatures above ~ 200 °C the WODB appears to accelerate the rate of bitumen oxidation. Air blowing at 240 °C can be used to produce an acceptable binder from blends of WODB and a softer grade of bitumen. The rate of hardening during air blowing depends on both the batch and concentration of WODB.

1. Introduction

The purpose of this study was to investigate the potential for use of vacuum distillation bottoms produced during the rerefining of waste lubricating oils, as extenders for roading bitumens.

In New Zealand these bituminous residues have little or no use, and the problem of disposal affects the economics of the whole rerefining process. Dumping is wasteful and environmentally unsound, and the high levels of lead and other wear metals in the residues prohibit their use as a fuel as combustion would release these pollutants to the atmosphere unless special precautions were taken. A typical analysis [1] of the residues used in the present study shows lead (29 000 mg kg^{-1}), magnesium (1000 mg kg^{-1}), chromium (46 mg kg^{-1}), cadmium (5.6 mg kg^{-1}), nickel (14 mg kg^{-1}), vanadium (1.1 mg kg^{-1}) and iron (1100 mg kg^{-1}). In Europe and the USA considerable quantities of distillation bottoms from rerefining operations are used as extenders of industrial and roofing bitumens.

The equivalent application in roading is not widespread or at least has not been widely reported in the literature [2, 3]. Mohammed *et al.* [4] have reported that paving-grade bitumen (40/50 pen) may be produced by blending 575 °C and 550 °C vacuum distillation residues from waste lubricating oils, at 10% concentration with a pen 6 base bitumen. This observation, however, is based only on softening point and penetration data.

The present work is a preliminary study to investigate more fully the simple rheological properties of waste oil distillation bottoms (WODB) and both simple and air-blown blends with bitumen. Results are compared with those for a standard unmodified roading bitumen. In addition, the oxidative properties of the extended bitumen were studied. Two accelerated ageing procedures–the rolling thin-film oven (RFTO), and the Australian durability oven–were used. In the RFTO procedure, a thin film (~ 1 mm) of bitumen is exposed at 163 °C to a stream of air for 85 min. This is meant to simulate the degree of oxidation likely to occur during asphalt manufacture. The durability oven procedure [5] was developed by the Australian Road Research Board and simulates long-term in-service ageing [6, 7]. The bitumen is first subjected to the RFTO procedure, and then aged at 100 °C in a stream of air until a "critical" viscosity associated with seal failure is reached.

2. Experimental procedure

2.1. Materials

The WODB used in this study are obtained from the vacuum distillation of waste automobile lubricating oils (370 °C, 3–6 mbar). The feedstock oil is first dried at 150 °C under a slight vacuum and then subjected to a proprietary chemical treatment, which helps prevent coking and fouling in the subsequent distillation step. This process does not involve acid or caustic treatment. The WODB are essentially neutral. The standard bitumen was 80/100 penetration grade, from a Safaniya Arabian crude. The 80/100 grade is produced by blending back straight-run 180/200 and air-blown 45/55 grades.

2.2. Physical properties

Viscosities at 70 and 135 °C, penetrations, softening points and flash points were determined using ASTM Methods D2171, D2170, D5, D36 and D92, respectively. Viscosities at 25 °C were determined using a cone and plate viscometer. Plots of log(shear rate) versus log (viscosity) were interpolated to give apparent viscosities at 0.05 s^{-1}.

Shear susceptibilities were calculated from cone and plate data using the power-law relationship [8], $\tau = \eta^c$, where τ = shear stress, γ = shear rate and c = index of complex flow. Over the range of shear rates used here, plots of $\ln \tau = \ln \eta + c \ln \gamma$ were linear and were used to calculate c. The value of c decreases as the non-Newtonian character increases.

Viscosity temperature susceptibility (VTS) was calculated using the usual definition [9], i.e. VTS = (log log η_{T_2} − log log η_{T_1})/(log T_1 − log T_2), where η_T = apparent viscosity at T_1 or T_2 (K). In the present work $T_1 = 70\,°\mathrm{C}$ and $T_2 = 135\,°\mathrm{C}$. For the purposes of the VTS calculation, it was considered sufficiently accurate to convert kinematic viscosities measured at 135 °C from centistokes to Pa/s assuming the density of the bitumen–WODB blends at 135 °C to be the same as that of the base bitumens.

2.3. Accelerated ageing methods

The two procedures used have been described above. The RFTO and durability oven procedures were carried out according to ASTM D2872 and Australian Standard Method AS 2341, respectively.

2.4. Air blowing

The blowing apparatus consisted of a stainless steel cylinder (150 mm i.d. × 250 mm height) surrounded by a high-temperature oil bath (Vitrea oil, Shell Chemicals NZ Ltd). The bath was mechanically stirred and heated by a 1000 W immersion heating element, controlled automatically to ± 1 °C by an Omron PID temperature controller and platinum resistance probe. The actual temperature of the charge was measured with a calibrated mercury thermometer. Air was bubbled through the charge by means of a 4 mm i.d. copper tubing spider with 14 (15 mm spaced) 1 mm diameter holes facing the bottom of the vessel. The sample was stirred with a two-bladed mechanical stirrer which extended to just above the bottom of the sample chamber. The air used was commercial-grade dry air and was controlled with a Matheson flowmeter. Following the practice of Giavarini and Saparito [10], a valve was installed to allow a rapid switch to an oxygen-free nitrogen line in case of emergency. This, however, was not needed and good temperature control was easily maintained.

A precisely weighed charge of about 2.2 kg was used in each experiment. This left about 9 cm headspace in the sample chamber. The blends were prepared as described previously and heated to about the blowing temperature in an oven just before blowing. Antifoam agent (~ 130 mg silicone F11 1/350, ICI NZ Ltd) was added and stirred in before the sample was introduced to the already heated blowing apparatus. The exact sample weight added was calculated by difference. Mechanical stirring was started at this time. As soon as the charge had reached the blowing temperature (normally 20–30 min) a weighed initial sample was taken and blowing began (the air flow rate being based on the recalculated charge weight). As samples were taken during the course of the experiment, the air flow rate was adjusted accordingly.

The blowing conditions used were as follows:

Charge weight:	~ 2.2 kg
Temperature:	240 ± 1 °C
Air flow rate:	1.4 l $\mathrm{min^{-1}\,kg^{-1}}$
Stirring rate:	10 r.p.m.

The conditions chosen are typical of those described in the literature [11–13] for laboratory-scale blowing units. The temperature is typical of that used industrially.

2.5. Thermogravimetric analysis

Measurements were made on a Mettler TG50 thermobalance/TC10A processor system. Ash contents of the WODB and blends were determined using samples of 10 ± 1 mg in a 0.15 $\mathrm{cm^3}$ platinum crucible. The temperature was increased from 35 to 650 °C at 15 °C $\mathrm{min^{-1}}$. The purge gas was air or nitrogen at 200 $\mathrm{cm^3\,min^{-1}}$. Weights were corrected for buoyancy effects using a blank run with an empty crucible. Duplicate analyses were made and the average reported to ± 0.5%, this being twice the pooled standard deviation of the duplicate analyses.

2.6. Compatibility of bitumen–WODB blends

Bitumen–WODB blends were stored in an airtight brass cylinder (50 mm i.d. × 150 mm height) fitted with

TABLE I Physical properties of various WODB batches

Batch	Data taken	Viscosity at 25 °C (Pa s)*	Viscosity at 70 °C (mPa s)	Viscosity at 135 °C (cst)	Ash (wt %)	Viscosity temperature susceptibility
DB1	8/8/89	31.30	69 200	2990	15.66	1.94
DB5	4/5/90	n.d.	34 400	1330	11.68	2.19
DB6	7/5/90	104.0	37 300	1490	11.81	2.14
DB7	15/5/90	n.d.	41 500	7857	12.03	1.01
DB8	22/5/90	n.d.	5560	376	10.60	2.21
DB9	30/5/90	196.0	3620	234	9.75	2.40
DB10	22/6/90	n.d.	34 600	1710	13.34	1.99
DB11	28/6/90	n.d.	39 300	2960	12.98	1.64
DB12	28/8/90	n.d.	14 800	898	12.63	2.03
DB13	11/9/90	n.d.	4050	315	9.40	2.17
80/100	–	643,000	55 200	425	0.14	3.45

* n.d. = not determined.

taps 15 mm from both the top and bottom. The cylinder was filled to ~ 10 mm above the upper outlet. After three days' undisturbed storage at 135 °C, samples were removed first from the upper outlet and then from the bottom. In both cases the first few millilitres were discarded to purge the dead volume of the taps.

3. Results

3.1. Properties of the waste oil distillation bottoms

The WODB are black, adhesive, viscous materials similar in appearance to conventional petroleum bitumens. To evaluate the consistency of the material, measurements were made on a number of batches collected over a period of about 13 months (Table I).

From Table I it is clear there is considerable variation in viscosity from batch to batch, which reflects variations in vacuum efficiency and feedstock. Although the WODB are of much lower viscosity than bitumen at 25 °C, the viscosities are comparable at 70°C and higher at 135 °C. The lower temperature susceptibility of the WODB compared to bitumen is also evident from the VTS values of around 2 compared to 3.45 for bitumen.

For safe handling and application, bitumens are normally required to have flash points of greater than ~ 220 °C; typically they are of the order of 300–350 °C. Flash points of a number of WODB batches were measured and found to be greater than 320 °C.

Thermogravimetric (TG) and differential TG (DTG) curves of 80/100 bitumen and a number of typical WODB batches are presented in Figs 1 and 2; weight losses as a percentage of the total weight lost are listed in Table II. The TG curves in the non-oxidizing nitrogen atmosphere are similar to that of bitumen, and show the onset of weight loss at a similar temperature (~ 250 °C). Even DB9, which has the lowest viscosity of the group, shows no significant low-temperature loss of volatiles. In fact, the rate of weight loss between 300 and 400 °C is less than that of the bitumen. The curves for different WODB under air are similar to one another, but quite different to that of the standard bitumen, especially at higher temperatures. This is most evident from the DTG curves where it is apparent that the WODB lack the 480–575 °C peak present in the bitumen curve. This is indicative of a significantly greater rate of oxidation compared to the bitumen. Both the WODB and bitumen curves show a number of sharp, non-reproducible peaks and spikes under air, but not under nitrogen. These are thought to be due to exothermic oxidation reactions producing localized and temporary surges in the rate of mass loss.

3.2. Properties of bitumen–WODB blends

Measurements were made using blends of 80/100 bitumen and a number of WODB samples including DB1 and DB9, which represented extremes in viscosity. Inspection of the results in Table III shows that

TABLE II Weight losses (as % of total weight lost) on heating

Material	Weight loss (%)									
	35–100 °C	100–200 °C	200–300 °C	300–400 °C	400–500 °C	500–600 °C	600–630 °C	600–700 °C	700–800 °C	800–900 °C
	Air atmosphere									
DB1	0.3	0.6	1.2	21.5	69.3	6.8				
DB5	0.3	0.3	1.1	19.1	72.0	6.7				
DB6	0.3	0.3	1.3	19.1	70.3	8.5				
DB8	0.2	0.3	1.5	21.9	66.9	8.8				
DB9	0.2	0.4	2.1	21.7	72.9	2.3				
DB10	0.2	0.4	1.7	22.7	71.5	3.2				
DB11	0.2	0.3	2.1	25.5	65.4	6.3				
DB12	0.3	0.6	1.8	24.1	70.0	2.8				
80/100	0.2	0.3	2.3	22.1	41.1	34.2				
	Nitrogen atmosphere									
DB1	0.4	1.0	1.2	12.5	71.2	3.6	0.3	2.2	6.6	1.4
DB5	0.4	1.1	1.1	15.7	68.7	3.6	0.4	5.6	2.8	0.9
DB6	0.4	0.9	1.0	15.6	69.5	3.4	0.4	5.8	1.9	2.0
DB8	0.3	0.4	1.1	14.97	70.9	4.9	0.3	4.5	2.2	0.8
DB9	0.2	0.4	1.2	18.4	68.0	3.7	0.3	5.2	1.8	1.0
DB10	0.5	1.3	1.8	20.4	63.1	3.6	0.3	6.3	1.6	1.4
DB11	0.4	0.9	1.4	19.8	64.6	2.8	0.3	2.7	5.9	1.4
DB12	0.4	1.3	1.5	20.0	64.5	3.4	0.3	6.1	1.6	1.2
80/100	0.2	0.3	2.4	17.8	52.6	7.3	0.0	14.2	5.1	2.5

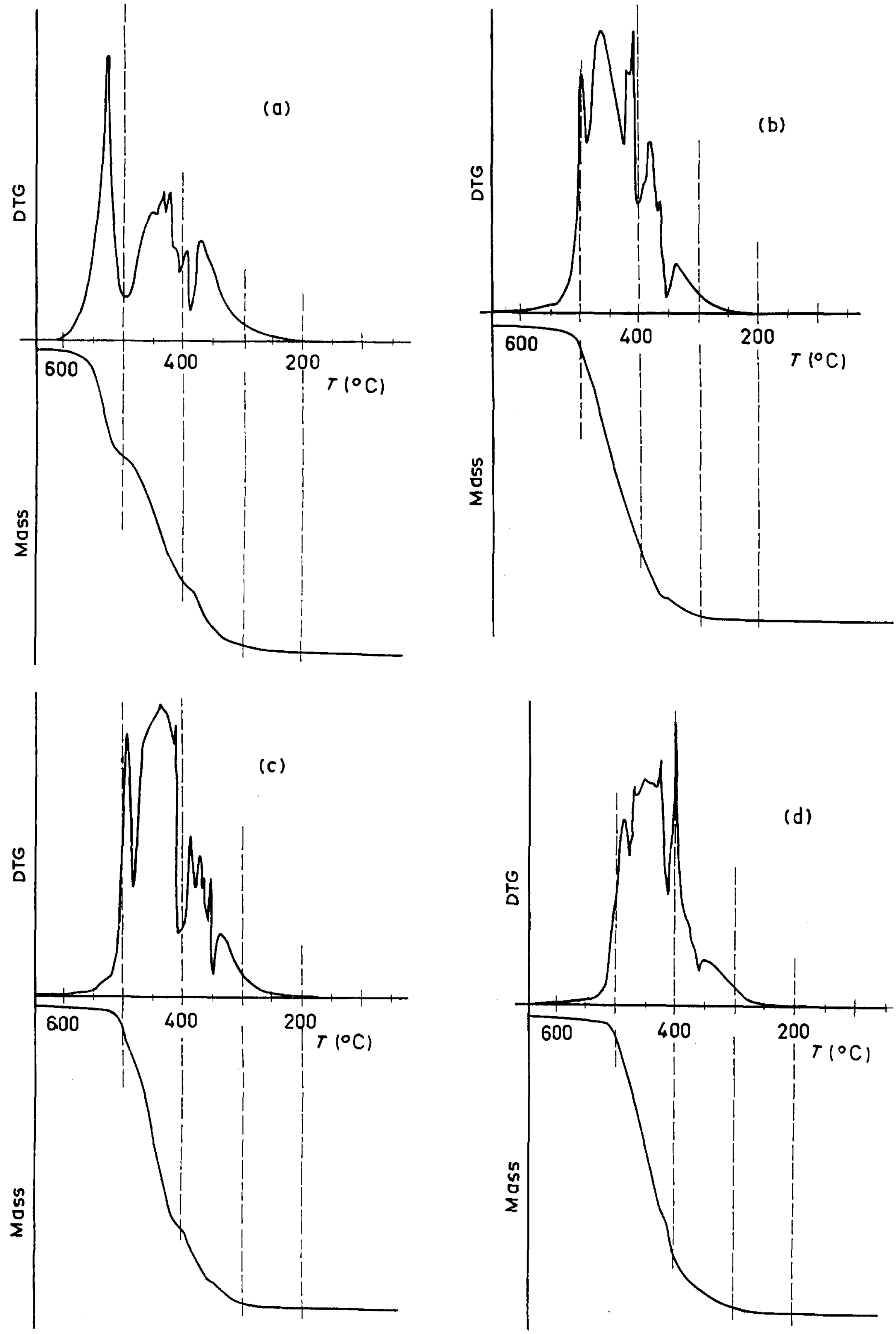

Figure 1 TG and DTG curves under air: (a) 80/100 bitumen, (b) DB1, (c) DB9, (d) DB11.

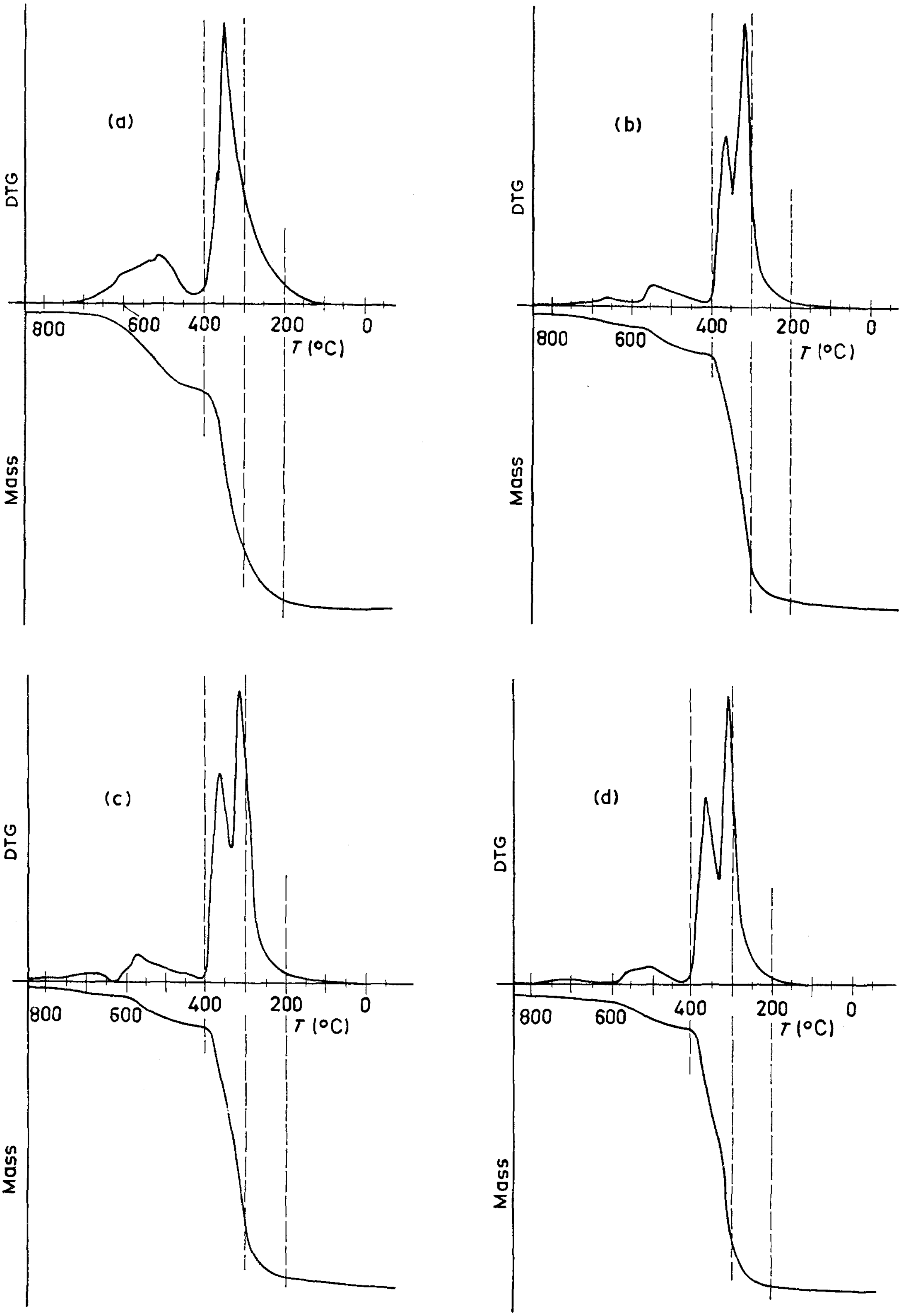

Figure 2 TG and DTG curves under nitrogen: (a) 80/100 bitumen, (b) DB1, (c) DB9, (d) DB11.

TABLE III Physical properties of bitumen–WODB blends

Blend (wt %)		Penetration at 5 °C (0.1 mm)	Penetration at 25 °C (0.1 mm)	Softening point (°C)	Viscosity at 70 °C (mPa s)	Viscosity at 135 °C (cst)	Viscosity temperature susceptibility	Solubility in trichloroethylene (wt %)
80/100 bitumen		14	91	48.3	55 200	425	3.45	99.88
80/100 + DB1	2%	13	97	48.1	53 800	431	3.42	n.d.
	4%	15	97	47.2	55 800	440	3.42	99.72
	10%	17	111	46.9	52 000	454	3.35	n.d.
	15%	18	148	45.4	50 600	464	3.32	n.d.
	20%	22	154	45.7	50 800	479	3.30	98.24
80/100 + DB9	4%	18	105	47.2	52 100	423	3.42	99.67
	10%	25	126	44.7	43 300	392	3.40	n.d.
	20%	30	167	41.4	42 400	384	3.41	98.40
80/100 + DB6	10%	18	112	46.3	52 700	436	3.40	n.d.
80/100 + DB7	10%	19	108	47.8	58 200	455	3.41	n.d.
80/100 + DB11	10%	21	118	46.1	51 800	455	3.35	n.d.

the effect of DB1 was to soften the bitumen except at 135 °C where the viscosity increased. The other blends, however, were softer at all temperatures including 135 °C compared to the base bitumen. These observations suggest that complex interactions are occurring between the WODB and the bitumens. For instance, the 70 °C viscosity (Table I) of DB1 is 69 200 mPa s and that of 80/100 bitumen 55 200 mPa s, yet on mixing the blend becomes softer than either constituent, having a 70 °C viscosity of around 51 000 mPa s, even at high concentrations of DB1. Conversely, the 135 °C viscosity of the same blends increases, as seems reasonable from the 135 °C viscosities of the constituents.

Over the range studied the observed effects were approximately linear with increasing WODB concentration, and for all blends the effect of a given concentration on the base bitumen properties were greater, relatively speaking, at low temperatures (5 and 25 °C) than at 70 or 135 °C. Hence the 10% blends produce an average change of ~ 40% in the 5 °C penetration value of the 80/100 bitumen, but only ~ 6% in the 135 °C viscosity. The softening of the bitumen at 5 °C and the overall trend towards a reduction in temperature susceptibility (as indicated by the VTS) is, in general, desirable for a roading bitumen. The variability of the WODB is also apparent in the blends. Hence, at the 10% level the changes in, for example, the 25 °C penetration range from 18% for DB7 to 38% for DB9. The solubility in trichloroethylene of the blends decreased with concentration of WODB and the decrease is of a magnitude consistent with the ash contents of the original WODB.

In practical applications it would be necessary to store bitumen–WODB blends at elevated temperatures for extended periods. It was thus important to investigate any tendency of the materials to separate on storage or for any phase separation of the blend as a whole. Experiments were carried out with blends stored at 135 °C for three days using DB1. As well as viscosity measurements, ash contents were measured as a good linear correlation ($R^2 = 99.8\%$) was found with DB1 concentration. The results (Table IV) show no significant separation of the two constituents. The viscosities and calculated concentrations of all samples are within error of one another and show no overall trend. This conclusion is in agreement with general observations made during the course of the study during which no problems with compatibility were apparent.

TABLE IV Bitumen–WODB blend compatibility

Sample	80/100 + DB1	
	Viscosity (Pa s) ± 8%	DB1 concentration (wt %) ± 1%
Initial	1.1×10^4	12.6
Top after 73 h at 135 °C	1.1×10^4	13.8
Bottom after 72 h at 135 °C	1.3×10^4	13.8
Remixed after 72 h at 135 °C	1.1×10^4	13.2

The accelerated oxidative hardening properties of a number of 10% blends are reported in Table V. The data indicate that the blends are hardening at a similar but somewhat slower rate to the base bitumen. The retained penetration for both the blends and the bitumen are similar, but the observed increases in viscosity as indicated by the η_R/η_0 ratios are considerably less for most of the blends. The blends also tend to have slightly higher durability oven values, but this may in part reflect their slightly lower initial viscosities. Differences in the degree of oxidation of a bitumen will also be apparent from measurement of shear susceptibility [14]. This is due to the fact that oxidation introduces polar functionalities such as carbonyl and sulphoxide groups which lead to a more highly structured material. As can be seen from Table V, all the materials show a slightly decreased (more shear-susceptible) c value after the RFTO treatment as is to be expected. However, the effect is similar in magnitude for both the bitumen and the WODB blends, as the c_0/c_r ratios are within error of one another.

Although further work is needed to confirm this point, the results in Table V suggest that at 163 °C the WODB may actually slightly improve the oxidative stability of the base bitumen; at the very least the WODB have no significant deleterious effect. However, at higher temperatures there is some evidence to the contrary. Thermogravimetric analysis of blends of

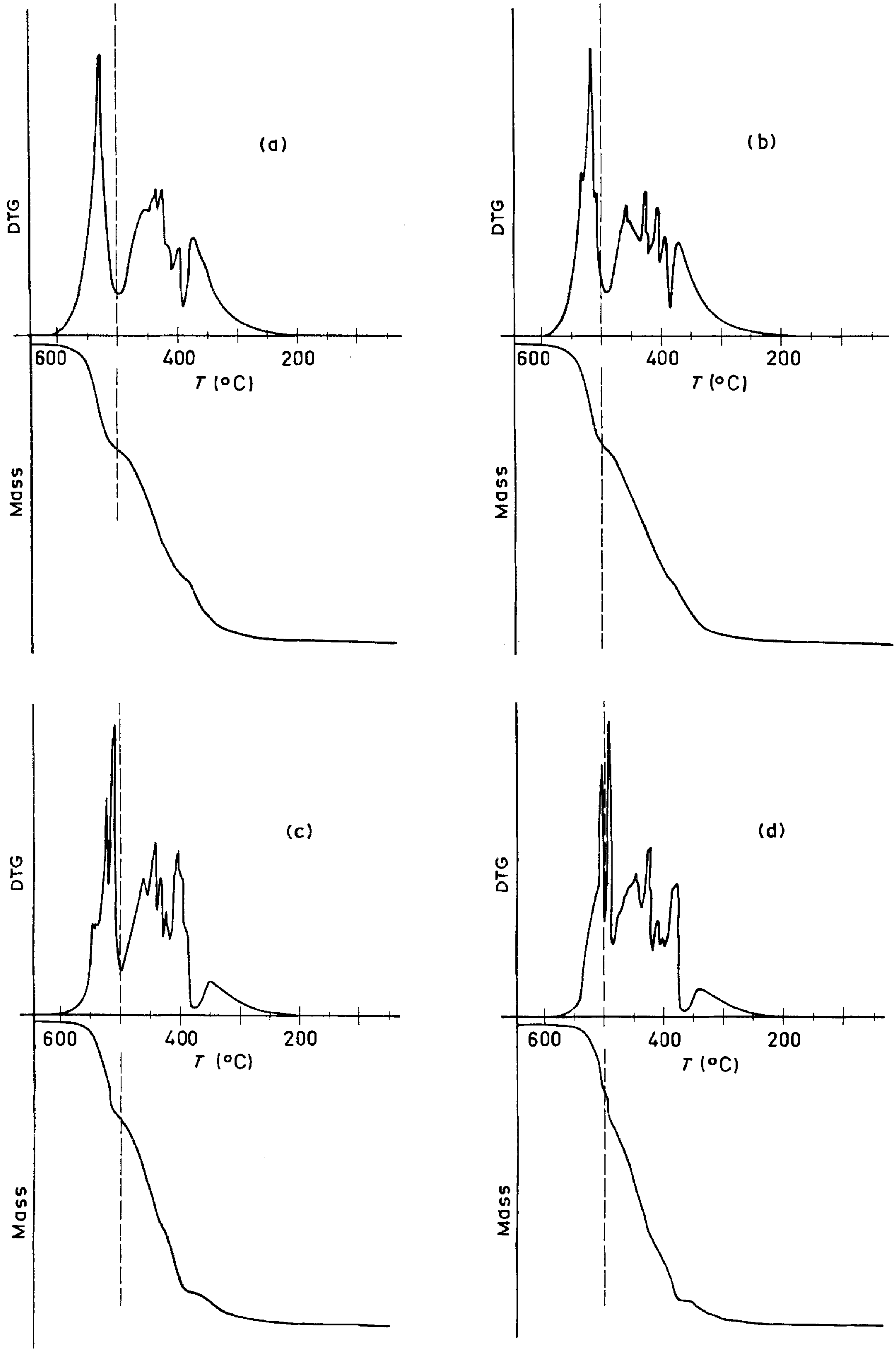

Figure 3 TG and DTG curves of 80/100 Bitumen–DB1 blends under air: (a) 80/100 bitumen, (b) 2% DB1, (c) 4% DB1, (d) 10% DB1, (e) 20% DB1.

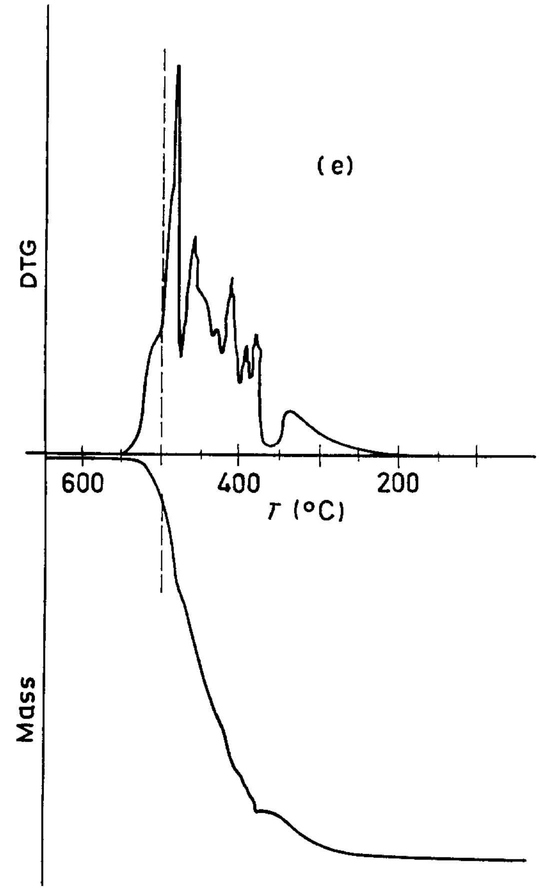

Figure 3 Continued

varying concentrations of DB1 in 80/100 bitumen in an oxidizing (air) atmosphere (Fig. 3) show a definite trend. The TG curve of 80/100 bitumen (Fig. 1) shows three major weight loss steps: 250–380 °C, 380–480 °C and 480–580 °C, with three corresponding peaks in the DTG. While only these steps are apparent in the 2% DB1 blends, the 5% blend shows resolution of the middle step into two, which reflects the presence of the WODB. At higher concentrations the third step becomes less distinct from the second. Most significantly, the temperature at which oxidative degradation is complete has dropped from 580 °C (2%) to 540 °C (20%). The effect is more apparent from inspection of the DTG curves, where the last major peak moves from around 530 °C (2% and 80/100 bitumen) to about 480 °C for the 20% blend.

3.3. Air blowing of bitumen–WODB blends

Results of air blowing experiments using blends of WODB in 180/200 bitumen are presented in Figs 4–6. The usual quantitative measure of reaction rate during air blowing is the rate of change of softening point ($\Delta SP\,h^{-1}$). Although Lockwood [15] and others [16] have found an exponential relationship between softening point and time, this was not observed in the present case, where with one exception (10% DB7) very good linear correlations were found. The slopes of the softening point plots in Fig. 4 are given in Table VI; in the case of DB7, the value given is the average over the time of the experiment. All of the blends

TABLE V Properties of 10% bitumen–WODB blends after RFTO and durability oven treatment

Blend	Penetration at 25 °C (0.1 mm)	% of pretreatment penetration retained (0.1 mm)	Viscosity at 25 °C (Pa s)		η_R/η_O (± 2)*	Complex flow		c_O/c_R (± 0.1)*	Durability (days)
			Before RFTO (η_O)	After RFTO (η_R)		Before RFTO (c_O)	After RFTO (c_R)		
80/100 bitumen	55.5	61	138 900	621 000	4.5	0.867	0.791	1.10	4.3
80/100 + DB1	73	64	148 000	510 000	3.5	0.807	0.707	1.14	6.7
DB6	76	68	139 000	446 000	3.2	0.863	0.681	1.27	5.6
DB9	78	62	107 000	348 000	3.3	0.836	0.707	1.18	7.6
DB11	71	60	103 000	437 000	4.2	0.827	0.744	1.11	n.d.

* The quoted errors are twice the pooled standard deviation for replicate analyses.

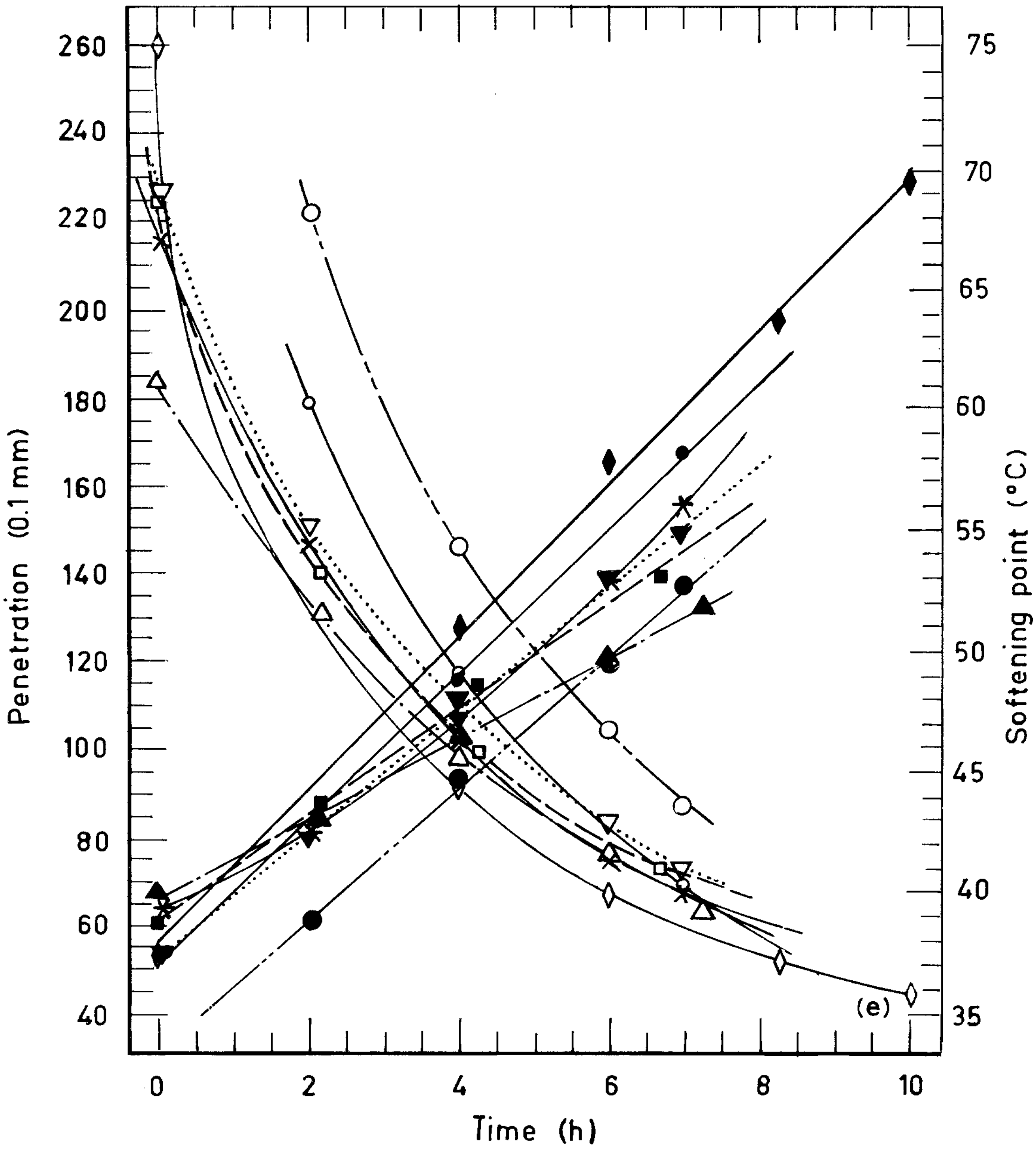

Figure 4 Effect of blowing time on penetration at 25 °C (open symbols) and softening point (filled symbols) of 180/200 bitumen–WODB blends: (△, ▲, —·—) 180/200; (□, ■, ——) 10% DB6; (×, *, ——) 10% DB7; (▽, ▼, ···) 10% DB11; (◇, ◆, ——) 20% DB6; (○, ●, – – – –) 20% DB9; (○, ●, ——) 20% DB11.

TABLE VI Rate of oxidative hardening of air-blown bitumen–WODB blends

Blend (wt %)	ΔSP h^{-1} (°C h^{-1})
180/200	1.67
180/200 + DB6 10%	2.18
DB7 10%	2.50
DB11 10%	2.41
DB6 20%	3.15
DB11 20%	2.96
DB9 20%	2.70

harden at a faster rate than the 180/200 bitumen. The rate also varies with WODB batch and increases with the concentration of a particular WODB blend. The variation in hardening rate from batch to batch is also very apparent from the 135 and 70 °C viscosity curves (Figs 5 and 6). Hardening due to loss of volatile components can be largely discounted, as the design of the blowing still produced essentially reflux conditions.

To compare the blown products with the standard 80/100 bitumen, the penetration curves in Fig. 4 were interpolated to give the time required to reach a penetration of 90. These times were then used to calculate softening point and viscosities from Figs 4–6 (Table VII). It is evident that air blowing of bitumen–WODB blends to the approximate equivalent of an 80/100 bitumen is quite feasible, especially at 10% concentration. However, it is also evident that the different rates of oxidative hardening amongst the blends means that the blowing process does not, as hoped, remove the initial variation in their consistency; in fact it tends to accentuate it. For example, the difference in initial 135 °C viscosities of the 10% DB7 and DB11 blends is 13 mPa s, but after 7 h of

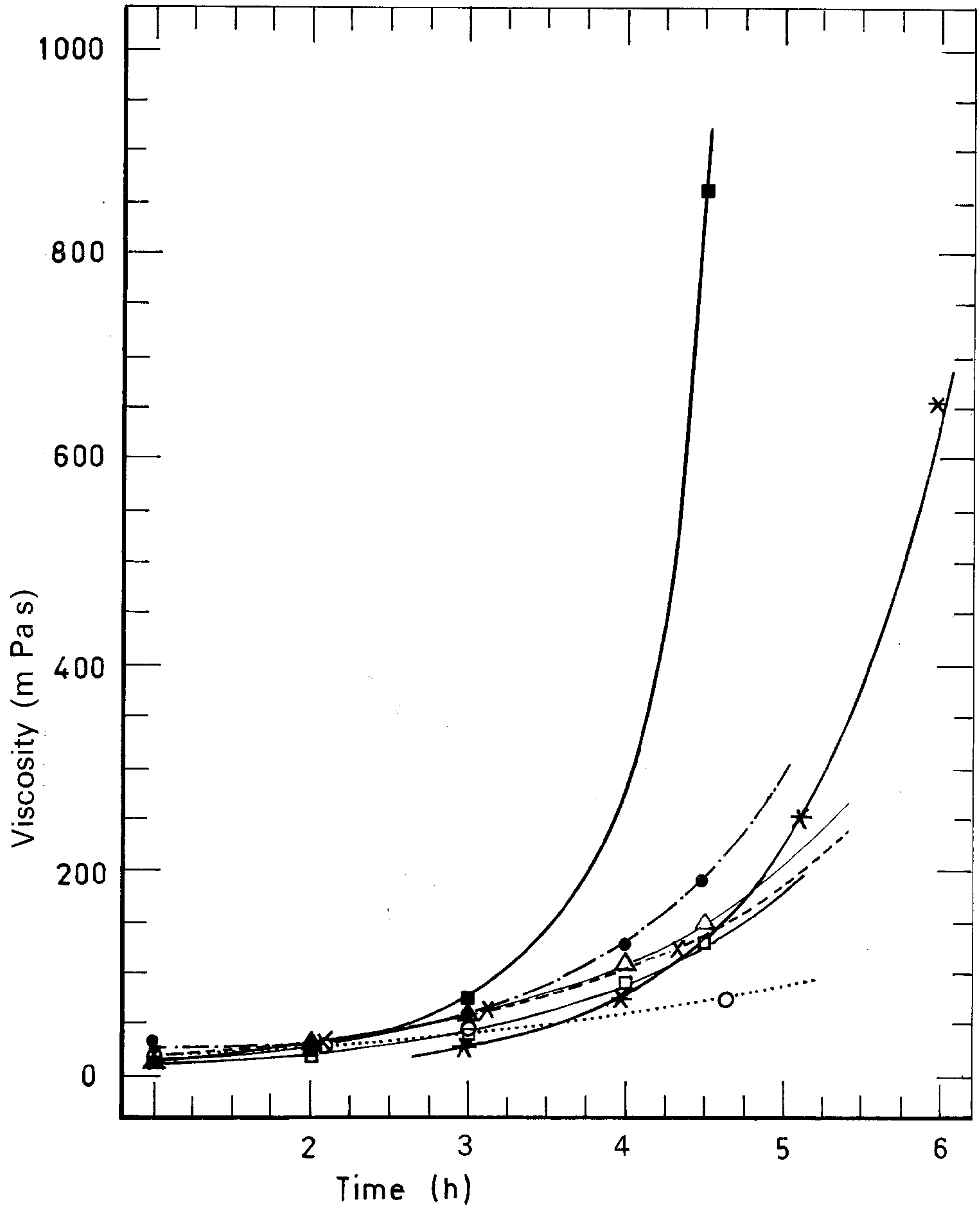

Figure 5 Effect of blowing time on viscosity at 70 °C of 180/200 bitumen–WODB blends: (○, ···) 180/200; (×, – – –) 10% DB6; (●, —·—) 10% DB7; (△, ——) 10% DB11; (∗, ——) 20% DB6; (□, ——) 20% DB9; (■, ——) 20% DB11.

TABLE VII Physical properties of air-blown bitumen–WODB blends interpolated to a penetration (25 °C) of 90

Blend	Time required to reach Pen 90 (h)	Penetration at 25 °C (0.1 mm)	Softening point (°C)	Viscosity at 70 °C (mPa s)	Viscosity at 135 °C (cst)	Viscosity temperature susceptibility
80/100	–	91	48.3	55 200	425	3.45
180/200	4.792	90	47.7	48 600	433	3.36
180/200 + DB6 10%	4.923	90	49.6	76 000	507	3.45
DB6 20%	4.118	90	51.7	119 000	608	3.51
DB6 50%	5.680	90	–	2 432 000	3220	3.49
DB7 10%	4.781	90	50.7	62 300	586	3.21
DB9 20%	6.911	90	52.3	121 800	672	3.43
DB11 10%	5.396	90	51.0	100 000	581	3.41
DB20 20%	5.443	90	53.3	196 000	719	3.60

blowing it has increased to 67 mPa s. An approximately constant rate of hardening for different WODB batches would have allowed simple prediction of the required blowing time from the initial blend softening point.

4. Discussion

This preliminary study has shown that although the effects of the WODB on the physical properties of roading bitumen are complex, they are not so great as to preclude the use of viable practical concentrations

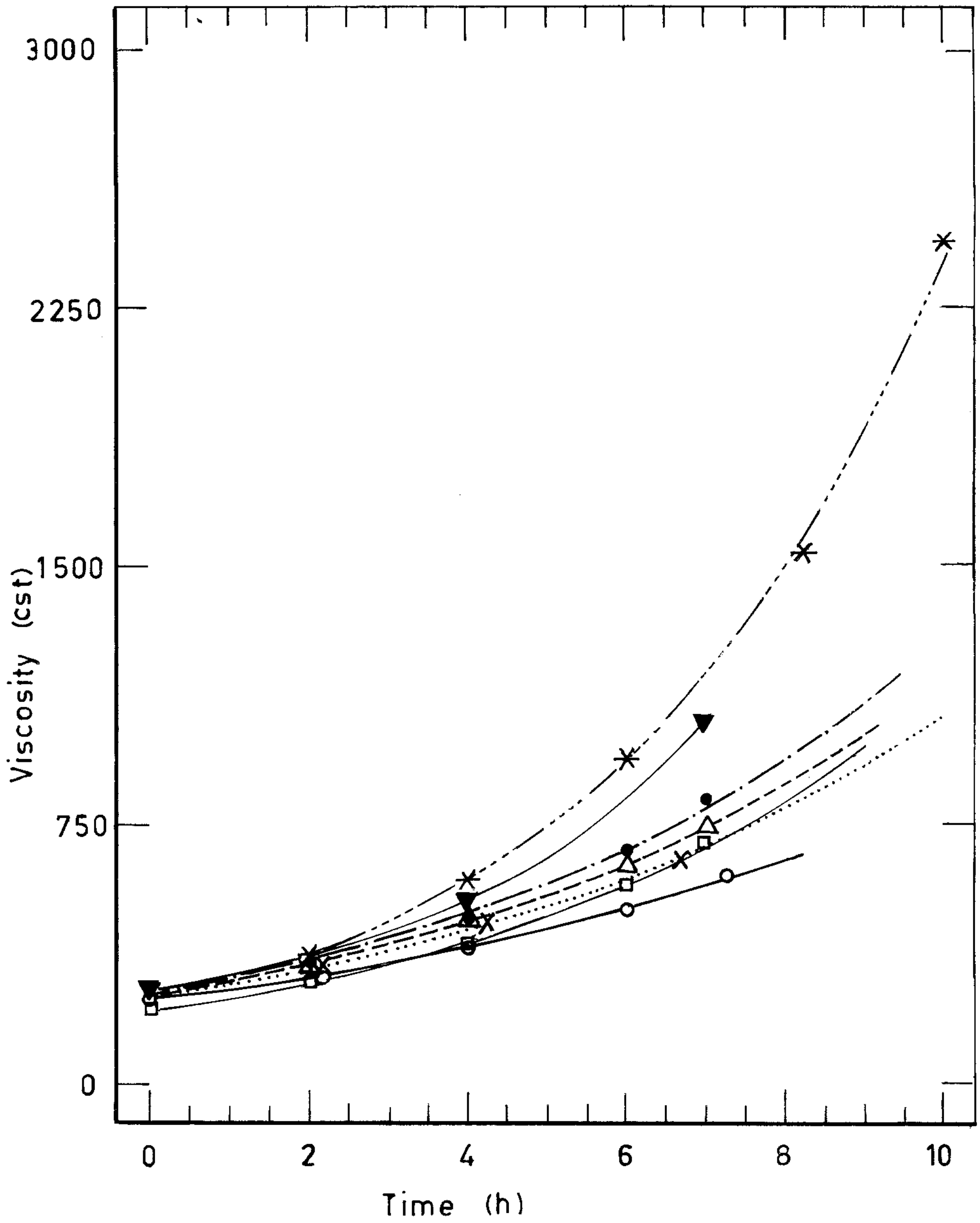

Figure 6 Effect of blowing time on viscosity at 135 °C of 180/200 bitumen–WODB blends: (○, ——) 180/200; (×, · · ·) 10% DB6; (●, —·—) 10% DB7; (△, — —) 10% DB11; (*, — - - - —) 20% DB6; (□, ——) 20% DB9; (▼, ——) 20% DB11.

(~ 10%) as bitumen extenders. In fact the trend towards lower temperature susceptibilities with increasing concentration of WODB is highly desirable in a roading bitumen, as long as the viscosity at handling and application temperatures is not too high. The variation in bitumen–WODB blend properties with WODB batch at the same concentration may pose difficulties in terms of meeting roading authority specifications. Although air blowing does allow better control of the blend properties, in practice variations in rate of hardening amongst the blends complicates the prediction of required blowing times to reach a required penetration or viscosity.

The oxidative hardening properties of the blends are similar to one another and to those of a standard bitumen, at least up to the highest temperatures likely to be experienced in practice (~ 200 °C). At higher temperatures the blends oxidize at a faster rate than bitumen. The oxidation-promoting effect of the WODB is possibly due to the high levels of wear metals present in the residues. Metals and their oxides are well known catalysts in the auto-oxidation reactions, contributing to the degradation of polymers [17] and hydrocarbons. Varying levels of metals may also help account for the variation in rates of hardening observed with different WODB batches during air blowing. There is, however, no simple correlation between crude ash content and ΔSP from Table VI.

To demonstrate the feasibility of manufacturing and laying asphalt using WODB-extended bitumen, a full-scale field trial has been carried out. Conventional equipment and techniques were used with 10 and 20% air-blown blends without difficulty or any noticeable differences compared to a control 80/100 bitumen. Details of the trial will be reported elsewhere.

The greatest difficulty with the WODB in terms of potential practical application arises from the variability in physical properties of the residues. This is principally a reflection of plant efficiency and is likely to continue to be a feature of the residues while they remain a low-value by-product of waste oil rerefining.

The results of this study suggest that further work, including a more sophisticated rheological investigation and study of the occupational health aspects of this type of material, is well warranted.

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References

1. J. O'CONNOR, (Dominion Oil Refining Co. Ltd), private communication (1991).
2. J. H. KIM, H. K. KANG and D. S. HERH, *Komu Hakhoechi* **20** (1985) 115.
3. J. W. SWAIN, Report No. EPA-IWS-144C, (Environmental Protection Agency, Washington, DC, 1980).
4. A. K. ABDUL HALIM MOHAMMED, K. HANKISH, A. R. NAGI, N. ABBAS and M. ABDUL-HUSSEIN SAID, *J. Pet. Res.* **3** (1984) 15.
5. E. J. DICKINSON, in Proceedings of 11th ARRB Conference, (Melbourne, Australia, 1982) p. 35.
6. J. W. H. OLIVER, *Transp. Res. Rec.* No. 1106 (1987) 196.
7. *Idem, Aust. Rd Res.* **20** (1990) 1.
8. K. MAJIDZADEH and H. E. SCHWEYER, *Proc. AAPT* **34** (1965) 20.
9. G. R. DOBSON, *J. Inst. Pet.* **58** (1972) 14.
10. C. GIAVARINI and S. SAPORITO, *Fuel* **68** (1989) 943.
11. L. W. CORBETT, *Ind-Eng. Chem. Process. Des. Dev.* **14** (1975) 181.
12. J. S. BAHL and H. SINGH, *Rev. Inst. Franc. Petrole* **38** (1983) 413.
13. M. A. QUDDUS and F. KHAN, *Fuel* **69** (1990) 509.
14. P. S. KANDHAL, L. D. SANDVIE and M. E. WENGER, *Proc. AAPT* **42** (1973) 99.
15. D. C. LOCKWOOD, *Pet. Ref.* **38** (1959) 197.
16. S. R. PATWARDHAN and S.B. KHADE, *Ind. Eng. Chem. Process. Des. Dev.* **21** (1982) 154.
17. B. RANBY and J. F. RABEK, "Photodegradation, Photo-Oxidation and Photo-Stabilisation of Polymers" (Wiley, London, 1975) p. 104.

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