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(54) Titre : AGENT D'ADOUCISSAGE SERVANT AU RECYCLAGE DE REVETEMENT DE BITUME
(54) Title: SOFTENING AGENTS FOR RECYCLING ASPHALT PAVEMENT

(57) Abrégé/Abstract:
Methods and compositions are provided for rejuvenating aged asphaltic paving material, or for improving the compatibility of aged asphalt in salvaged old asphalt paving materials with other asphaltic materials, or of improving the resilience to stripping of rejuvenated asphalt and aggregates, by the use of an agent which was heretofore not known to be a rejuvenating agent. Such rejuvenating agent is a sewage sludge-derived oil, or a fraction thereof, having a particularly recited composition. This rejuvenating agent markedly lowers the viscosity of the aged asphalt, disperses the components of the aged asphaltic paving material for dispersion of such, or other added asphaltic components in the asphalt, and increases the adhesion of aged asphaltic paving material to aggregates. Such rejuvenating agent may be used together with other conventional rejuvenating agents and/or softening agents, e.g., hydrocarbon products with physical characteristics which are selected to restore aged asphalt to the requirement of current asphalt specifications, for example, soft virgin asphalts.

ABSTRACT OF THE DISCLOSURE

Methods and compositions are provided for rejuvenating aged asphaltic paving material, or for improving the compatibility of aged asphalt in salvaged old asphalt paving materials with other asphaltic materials, or of improving the resilience to stripping of rejuvenated asphalt and aggregates, by the use of an agent which was heretofore not known to be a rejuvenating agent. Such rejuvenating agent is a sewage sludge-derived oil, or a fraction thereof, having a particularly recited composition. This rejuvenating agent markedly lowers the viscosity of the aged asphalt, disperses the components of the aged asphaltic paving material for dispersion of such, or other added asphaltic components in the asphalt, and increases the adhesion of aged asphaltic paving material to aggregates. Such rejuvenating agent may be used together with other conventional rejuvenating agents and/or softening agents, e.g., hydrocarbon products with physical characteristics which are selected to restore aged asphalt to the requirement of current asphalt specifications, for example, soft virgin asphalts.

(a) TITLE OF THE INVENTION

SOFTENING AGENTS FOR RECYCLING ASPHALT PAVEMENT

(b) TECHNICAL FIELD TO WHICH THE INVENTION BELONGS

This invention relates to the rejuvenation of aged asphaltic pavement.

(c) BACKGROUND ART

As asphaltic concrete pavement ages, the asphalt cement becomes harder. This leads to the deterioration of the pavement. During this age hardening the heavy asphaltic component contents increase. A rejuvenation agent for such aged asphaltic concrete must be of sufficiently low viscosity to lower the viscosity of the aged asphalt to desired levels. It also must have a high polar to non-polar component ratio to assure compatibility with the high heavy asphaltic content in the asphalt of the salvaged aged asphaltic pavement.

Recycling of salvaged asphalt pavement has significant economic advantages because significant amounts of virgin asphalt and aggregates are needed to produce a new asphaltic cement pavement. Further, salvaged asphalt pavement contains high quality aggregates which may be either disposed of in dump sites or may be used in low performance applications.

The current situation in North America concerning the hot recycle of salvaged asphalt pavement is quite extensively described in a paper by W. D. Robertson et al entitled "Mix Design - The Key to Successful Pavement Recycling" and published in the Canadian Technical Asphalt Association Proceedings of 1988, p. 330. A copy of that paper is attached hereto as Appendix M.

One common practice at this time is to mix old milled asphalt pavement material with soft virgin asphalt and then to heat the whole mixture by the addition of preheated virgin aggregate. The current limit in Ontario, Canada of old pavement utilization is 50%, due to environmental regulations for the amount of smoke produced when the hot aggregate is added.

Specifications for recycling agents used in hot-mix recycling are described in a paper by Kari et al in "Asphalt Paving Technology" of 1980 p. 177. A copy of that paper is attached hereto as Appendix N.

Cold in place recycling of asphalt pavements is increasing in popularity, as described by Wood et al in "Transportation Research Record" #1178 of 1988. A copy of that paper is attached hereto as Appendix O.

N. Paul Khosla described the use of emulsified recycling agents in "Asphalt Paving Technology" of 1982 p. 522. A copy of that paper is attached hereto as Appendix P.

However, other approaches to recycling are possible. These include methods that require minimal or no heating. Several approaches are described in the following patents:

U. S. Patent No. 4,373,961, patented February 15th, 1983 by E. M. Stone, provided a process and composition for recycling old asphalt pavements into new pavements. It was accomplished by adding a special asphalt emulsion to the crushed old asphaltic pavement material without requiring the addition of new aggregate or heating. The old material was then cemented at the ambient temperature into a new durable composition of high stability and water resistant, softening or other appreciable change in the old asphalt, by selection of the degree of hardness and character of the asphalt base stock in the special emulsion. Thus the patentee provided a process for recycling old asphalt pavements into new pavements comprising the steps of crushing the old asphalt pavement to form an aggregate comprising asphalt and mineral matter and then emulsifying an asphalt base stock with water to form an emulsion. The aggregate was then mixed with the emulsion to form a pavement material. The pavement material was then laid and compacted as a pavement course.

Other procedures in the prior art attempted to solve the problem by improving the asphalt compositions. Thus, U.S. Patent No. 2,904,494, patented September 15th, 1959 by R. L. Griffin, provided an improvement in the process of preparing asphalts having improved aging stability. The patented steps included distilling a petroleum residue to provide a reduced residue having improved stability. That residue was then blended with an asphaltic oil.

Other patents have attempted to solve the problem by adding various agents to asphalt paving compositions. Thus, U.S. Patent No. 3,793,189, patented February 19th, 1974 by L. W. Corbett, provided asphaltic compositions suitable for use in paving formulations and having desirable viscosity, temperature susceptibility and ductility properties by combining propane-precipitated asphalt, asphaltenes and a liquid petroleum derivative, e.g., a heavy distillate or residual oil.

Other patents were directed to reconditioning bituminous pavements. Thus, U.S. Patent No. 3,221,615, patented December 7th, 1965 by E. W. McGovern, provided for the revitalization of bituminous pavements which were in the process of becoming or which have become dry and brittle through aging and weathering by applying thereto a composition which was derived from coal tar and comprised a mixture of di-, tri- and tetracyclic aromatic compounds and their alkyl homologs containing lower alkyl groups together with a significant amount of phenolic and hydroxy derivatives.

U.S. Patent No. 4,278,469, patented July 14th, 1981, by T. Y. Yan et al, provided a ductile asphaltic composition adapted for repairing and surfacing distressed asphalt pavements which comprised a blend of an asphalt component selected from marginal asphalt materials, a highly aromatic petroleum refinery residuum solvent component, and a polymeric component which is substantially asphalt-soluble.

U.S. Patent No. 4,279,660, patented July 21st, 1981, by I. Kamo et al, provided a process for the recovery and reutilization of materials in existing asphalt pavements, comprising comminuting existing asphalt pavement, subjecting the comminuted pieces to the action of a solvent in a dissolving zone to separate asphalt from other component materials, recovering solvent and asphalt dissolved therein from the zone separately from the other materials, drying and classifying the other material according to size, separating asphalt from the solvent, and transferring separated asphalt and the size-classified other materials to storage zones for subsequent reuse in the preparation of asphalt pavement.

U.S. Patent No. 4,325,738, patented April 20th, 1982, by H. Plancher, provided a technique for substantially improving the useful life of asphalts by adding a minor amount of a moisture-damage-inhibiting agent selected from compounds having a pyridine moiety, including acid salts of such compounds. A shale oil fraction was said to serve as the source of the improving agent and may simply be blended with conventional petroleum asphalts.

U.S. Patent No. 4,549,834, patented October 29th, 1985, by J. P. Allen, provided an asphaltic composition especially suitable for rejuvenating recycled asphalt-aggregate road compositions, consisting essentially of asphaltic oils, asphaltic resins, and asphaltic pitch.

In spite of these many proposals of the prior art, there remains a need for the reconstitution of the used materials of existing asphalt pavements to provide almost new materials to be used in the construction of new asphalt pavements.

An object of a principal aspect of this invention is the utilization of sewage sludge-derived oils for the rejuvenation of asphalt in salvaged asphalt paving materials and to allow recycling.

(d) DESCRIPTION OF THE INVENTION

By one broad aspect of this invention, an asphaltic composition consisting essentially of: comminuted aged asphaltic pavement material; an effective amount, from 2% to 15% by weight of a blend of an agent which is selected from the group consisting of a soft asphalt cement, a conventional asphalt cement, and a cutback asphalt, with a nitrogen-containing, adhesion-improving, anti-stripping agent comprising a sewage sludge-derived oil comprising a mixture of saturated aliphatic hydrocarbons, monoaromatic hydrocarbons, diaromatic hydrocarbons, polyaromatic hydrocarbons, polar compounds and basic, pyridine-soluble compounds, having the following elemental chemical composition: nitrogen, 3.42% to 5% by weight; oxygen, 5.8% to 6.9% by weight, sulphur, 0.3% to 0.8% by weight; hydrogen, 9.7% to 10.4%, and carbon, 76.9% to 79.8%.

By one variant thereof, the amount of the sewage sludge-derived oil blend is from 2% to 12% by weight.

By another variant thereof, the asphaltic composition is blended with soft virgin asphalt.

By another variant thereof, the asphaltic composition also includes at least one additional agent which is selected from the group consisting of other asphaltic materials, at least one rejuvenating agent and at least one softening agent.

By a variation of this aspect and the above variants thereof, the sewage sludge-derived oil has the following elemental composition: nitrogen, 3.42% to 4.95% by weight; oxygen, 5.84% to 6.89% by weight, sulphur, 0.34% to 0.83% by weight; hydrogen, 9.70% to 10.44%, and carbon, 76.92% to 79.76%.

By another broad aspect of this invention, a method is provided for the rejuvenation of aged salvaged asphaltic pavement material, the method comprising: incorporating, into the old asphaltic pavement, a sewage sludge-derived oil, or a fraction thereof, the sewage sludge-derived oil comprising a mixture of saturated aliphatic hydrocarbons, monoaromatic hydrocarbons, diaromatic hydrocarbons, polyaromatic hydrocarbons, polar compounds and basic, pyridine-soluble compounds, having the following elemental chemical composition: nitrogen, 3.42% to 5% by weight; oxygen, 5.8% to 6.9% by weight, sulphur, 0.3% to 0.8% by weight; hydrogen, 9.7% to 10.4%, and carbon, 76.9% to 79.8%.

By another broad aspect of this invention, a method is provided for improving the compatibility of aged asphalt in salvaged old asphalt paving materials with other asphaltic materials, the method comprising: incorporating, into the old asphaltic pavement and into the other asphaltic materials, and/or rejuvenating agents and/or suitable softening agents, a sewage sludge-derived oil, or a fraction thereof, the sewage sludge-derived oil comprising a mixture of saturated aliphatic hydrocarbons, monoaromatic hydrocarbons, diaromatic hydrocarbons, polyaromatic hydrocarbons, polar compounds and basic, pyridine-soluble compounds, having the following elemental chemical composition: nitrogen, 3.42% to 5% by weight; oxygen, 5.8% to 6.9% by weight, sulphur, 0.3% to 0.8% by weight; hydrogen, 9.7% to 10.4%, and carbon, 76.9% to 79.8%.

By yet another broad aspect of this invention, a method is provided for improving the resistance to stripping of rejuvenated asphalt cement aggregate comprising the utilization of a sewage sludge-derived oil, or a fraction thereof, the sewage sludge-derived oil comprising a mixture of saturated aliphatic hydrocarbons, monoaromatic hydrocarbons, diaromatic hydrocarbons, polyaromatic hydrocarbons, polar compounds and basic, pyridine-soluble compounds, having the following elemental chemical composition: nitrogen, 3.42% to 5% by weight; oxygen, 5.8% to 6.9% by weight, sulphur, 0.3% to 0.8% by weight; hydrogen, 9.7% to 10.4%, and carbon, 76.9% to 79.8%.

By one variant of such methods, the sewage sludge-derived oil has the following elemental composition: nitrogen, 3.42% to 4.95% by weight; oxygen, 5.84% to 6.89% by weight, sulphur, 0.34% to 0.83% by weight; hydrogen, 9.70% to 10.44%, and carbon, 76.92% to 79.76%.

By another variant of such methods and variant, the amount of the sewage-derived oil is from 2 to 15% by weight.

The other agent, or agents, in the asphaltic composition may be a hydrocarbon product with physical characteristics which are selected to restore aged asphalt to the requirements of current asphalt specifications, e.g., a soft asphalt cement, a cutback asphalt, or a conventional asphalt cement; preferably it can be a soft virgin asphalt.

As noted above, the asphaltic composition may also include other asphaltic materials and/or rejuvenating agents and/or suitable softening agents. Such rejuvenating agents and/or suitable softening agents may be as described below:

a) A hydrocarbon product with physical characteristics selected to restore aged asphalt to the requirements of current asphalt specification, as described in Kari et al "Asphalt Paving Technology" 1980 p. 77, (Appendix N).

b) Soft asphalt cements and cutback asphalts. One example of such agents is that known by the Trade-mark MOBILSOL-30. Examples of other suitable modifiers are provided in Khosla, "Asphalt Paving Technology" 1982 page 522, (Appendix P).

c) Petroleum oils, soft asphaltic residues and conventional asphaltic cements, as described in Robertson et al " Mix Design-Key to Successful Pavement Recycling" page 330, (Appendix M).

The comminuted aged asphaltic pavement generally is of a size up to 1" in diameter.

Sewage, sludge-derived oils or fractions thereof are well suited to be used as asphaltic rejuvenation agents either neat or in combination with appropriate other conventional rejuvenating agents and/or softening agents for the following reasons:

1. They have low viscosities and thus they can soften the hardened asphalt by lowering viscosities and increasing penetration values.

2. They have an affinity for heavy residual components, e.g. asphaltenes. They are thereby compatible with asphalts of high asphaltene content and can also render other appropriate materials.

3. They improve resistance of rejuvenated recycled asphalt to stripping from the aggregates.

The sewage sludge-derived oil, unless topped at high temperatures, would not meet some of the specifications. However, it could be mixed with some other high boiling appropriate materials and then this mixture would then have the advantageous properties of both of these components for hot recycling.

In cold recycling, no complications are foreseen even if the +150°C sewage sludge-derived oil is used. In such case, the sewage sludge-derived oil could be used neat or together with other materials.

Such sewage sludge-derived oil may be produced according to the teachings of Canadian Patent No. 1,225,062, issued August 4th, 1987, to T. R. Bridle. The teachings of such patent may be summarized as follows:

A batch-type reaction system for the production of such sludge derived oil described in the above-identified Canadian patent may be operated as follows:

A single reactor provides both heating and reaction zones and consists of a PYREX tube of 70 mm diameter and 720 mm length. This was heated in a furnace, off-gases being condensed in a trapping system consisting of three series-connected flasks, using ice as the coolant. Non-condensable gases (NCG) were vented by pipe from the system to a furnace hood and not collected. A typical run was conducted by charging 550 g of dried sludge (93%-96% solids) into the reactor and deaerating with nitrogen while in the vertical position. The reactor volumetric packing for all runs was a nominal 50%. The reactor was then placed in the furnace, which was inclined by a support at $10^{o}C$ to facilitate liquid transport. All the lines, traps, etc. were connected and the entire system purged with nitrogen (15 mL/s) for 20 to 30 minutes. The furnace was then switched on and brought up to operating temperature at a controlled rate, the control employing a thermocouple placed in the sludge bed and connected to thermocouple switch and readout. Once operating temperature had been reached, the nitrogen purge rate was reduced to 7 mL/s. When all visible signs of reaction, i.e., gas/oil flow, ceased the heat was switched off and the nitrogen purge rate increased to 15 mL/s for approximately 30 minutes. The system was dismantled and the char, oil and pyrolytic water collected and stored for analysis, oil/water separation being achieved using a separatory funnel.

The operating conditions and results are shown in Table A below, while typical elemental analyses of the resultant oils and chars are shown in Table B and a distribution analysis of aliphatic hydrocarbons found in an oil is shown in Table C. All the data in the tables are expressed on a total solids basis (not corrected for volatiles). The non-condensable gas (NCG) yield was calculated by difference. Analysis of the NCG, by GC, indicated that it contained roughly 6% methane and 10% carbon monoxide with the remainder comprising mostly carbon dioxide and nitrogen. The calculated calorific value is approximately 2.0 MJ/kg of NCG.

Most of the test runs were conducted at optimum conditions defined as:

- optimum conversion temperature as determined by differential scanning calorimetry;
- linear increase of temperature with time to operating temperature at 10°C/minute; and
- continuous nitrogen purge.

Runs 11, 12, 13, 22, 24 and 19 instead were conducted with one variable altered during each test, as indicated in Table A.

Table A

TEST RUN CONDITIONS AND RESULTS

OPERATING CONDITIONS				RESULTS							
				OIL			CHAR				
Run No.	Sludge	Temp (°C)	Other Comments	Yield (%)	Cal.Value (MJ/kg)	Viscosity+ (Centistokes)	Yield (%)	Cal.Value (MJ/kg)	NonCondensable Yield(%)	Pyrolytic Water Yield(%)	Thermal Efficiency (%)
1,20,29	C	400	Optimum	20.8	36.40	Solid	59.5	9.86	11.6	8.1	81.9
5-10	D	450	Optimum	21.1	37.43	31.1	52.5	10.68	13.2	13.1	77.7
14,15,16	B	450	Optimum	24.1	33.13	60.5	53.7	10.08	13.3	8.8	83.2
2	C	425	63% WAS	25.8	33.83	70.3	57.1	11.35	12.2	4.9	87.2
3	C	425	75% WAS	28.6	34.13	97.5	56.7	11.63	10.1	4.6	90.8
4	C	425	88% WAS	28.7	31.77	214.0	54.6	10.65	8.9	7.8	82.4
11,12,13	B	350	Low temperature	12.8	33.32	Solid	65.6	12.00	10.3	11.2	79.3
22	C	450	High temperature	22.3	38.87	Solid	54.6	9.39	12.1	11.0	80.4
23	C	400	No N_2 purge during run	19.8	38.00	44.9	59.1	10.51	12.2	8.9	80.1
24	C	400	Ramp at 5°C/min.	16.3	37.92	Solid	62.7	11.24	10.3	10.7	76.9
19	C	400	10000 ppm Ni spike	20.9	33.98	63.4	60.8	NA	10.6	7.7	88.7
31	C	400	Second reactor, empty	19.0	37.49	Solid	60.0	11.07	12.0	9.0	80.1
32	C	400	Second reactor, char	17.2	38.18	39.5	59.9	11.07	13.0	9.9	77.0
33	C	400	Second reactor, catalyst	19.0	37.49	31.0	56.8	10.01	14.8	9.4	75.0

NA = Not Available

* Solid defined as 214 centistokes

+ Measured at room temperature (20-25°C) cf Table 5 measurement at 38°C (ASTM standard)

TABLE B

OIL AND CHAR ELEMENTAL ANALYSIS (%)

	OIL					CHAR				
Run No.	C	H	N	S	O	C	H	N	S	O
20	78.00	10.10	3.99	0.75	6.18	25.45	1.97	2.79	1.39	11.90
9	78.74	10.17	3.45	0.41	6.37	26.02	1.61	3.01	1.16	12.70
15	77.39	9.70	4.95	0.83	6.90	24.53	1.22	2.84	0.74	9.26
22	77.92	10.20	3.99	0.61	6.51	22.53	1.34	2.54	1.52	12.54
23	78.00	10.30	3.42	0.74	7.00	23.83	1.70	2.59	1.44	11.55
24	77.91	10.44	3.87	0.74	6.48	24.76	1.85	2.83	1.33	12.37
19	79.07	10.06	4.66	0.53	7.07	23.36	1.56	2.76	1.48	13.25
31	76.92	10.15	4.11	0.65	6.89	26.53	2.13	2.80	1.31	11.94
32	79.76	10.25	4.19	0.56	5.84	25.97	1.98	2.80	1.34	11.63
33	79.30	10.41	3.49	0.34	5.84	24.22	1.62	2.74	1.50	11.35

TABLE C

ALIPHATIC HYDROCARBON DISTRIBUTION IN OIL

Compound	%
C_{10}	8
C_{10-15}	30
C_{15-16}	6
C_{16-17}	5
C_{17-19}	10
C_{19-20}	10
C_{20-21}	10
C_{21}	21
	100

The above-described sewage sludge-derived oils may have the following composition:

Nitrogen	2% - 8%
Oxygen	3% - 10% (varies with degree of dehydration)
Sulphur	trace - 4%
Hydrogen	8% - 11+%

The sewage sludge-derived oils can be dehydrated by distillation. Large portions of the nitrogenous groups appear to be amine and amides with some pyridinic and pyrrolic types. Large portions of the oxygen containing groups appear to be carboxylic and amide types.

The following standard test methods were used for determining the properties of the rejuvenated old asphaltic paving materials containing sewage sludge-derived oil or fractions thereof:

The Standard Specification for Viscosity-Graded Asphalt Cement for Use in Pavement Construction is ASTM D3381-83. A copy of the description of that test is attached hereto as Appendix A.

The Standard Specification for Penetration-Graded Asphalt Cement for Use in Pavement Construction is ASTM D-946-82. A copy of the description of that test is attached hereto as Appendix B.

The Standard Test Method for Penetration of Bituminous Materials is ASTM D5-86. A copy of the description of that test is attached hereto as Appendix C.

The Standard Test Method for Softening Point of Bitumen (Ring-and-Ball Apparatus) is ASTM D 36-86. A copy of the description of that test is attached hereto as Appendix D.

The Standard Test Method for Effect of Heat and Air in Asphaltic Materials (Thin-Film Oven Test) is ASTM D1754-87. A copy of the description of that test is attached hereto as Appendix E.

The Standard Test Method for Ductility of Bituminous Materials is ASTM D 113-86. A copy of the description of that test is attached hereto as Appendix F.

The Standard Test Method for Solubility of Asphalt Materials in Trichloroethylene is ASTM D 2042-81. A copy of the description of that test is attached hereto as Appendix G.

Standard Test Method for Kinematic Viscosity of Asphalts (Bitumens) is ASTM D 2170-85. A copy of the description of that test is attached hereto as Appendix H.

Standard Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer is ASTM D 2171-88. A copy of the description of that test is attached hereto as Appendix I.

The Standard Test Method for Separation of Asphalt into Four Fractions is ASTM D4124-86. A copy of the description of that test is attached hereto as Appendix J.

The Standard Definitions of Terms Relating to Materials for Roads and Pavements is ASTM D8-88. A copy of such definitions is attached hereto Appendix K.

Method of Test For Stripping By Static Immersion is described by Ministry of Transportation of Ontario (MTO). A copy of the description of that test is attached hereto as Appendix L.

The materials used in the following examples were as follows:

Distillation fractions of sewage sludge-derived oils: +150°C, +250°C, +350°C, and +400°C; Shell, Gulf, and Petro Canada 85/100 Pen grade asphalt; Petro Canada 150/200 Pen grade asphalt; and Local (Ottawa, Canada area) milled recycled asphalt cement (RAC).

The sewage sludge-derived oils used have the compositions as previously described.

(e) DESCRIPTION OF THE FIGURES

In the accompanying drawings,

Figure 1 is a graph showing penetration (in dmm), as ordinate and Wt% sewage sludge-derived oil in recovered asphalt cement as abscissa;

Figure 2 is a graph of viscosity (in cSt) as ordinate and Wt% sewage sludge-derived oil in recovered asphalt cement as abscissa;

Figure 3 is a graph of penetration (PEN in PT 25°C) as ordinate and % sewage sludge-derived oil, +350°C additive, in aged asphalt as abscissa; and

Figure 4 is a bar graph of blends of asphalt, additive and aggregate showing the retaining coating.

As seen from the drawings, Figure 1 is a graphical summary of the penetration tests previously described showing the blend of recovered asphalt cement and sewage sludge-derived oil (+150°C), vs. penetration at 4°C and at 25°C.

Figure 2 is a graphical summary of the viscosity tests previously described showing the blend of recovered asphalt cement and sewage sludge-derived oil (+250°C), vs. viscosity at 100°C and at 135°C.

Figure 3 is a graphical summary of the penetration tests previously described showing the penetration vs. sewage sludge-derived oil, +350°C additive, blended with aged asphalt.

Figure 4 shows the amount of retained coating after the stripping by immersion test previously described.

The method of incorporation of sewage sludge-derived oils into salvaged asphaltic pavement for recycle can be by various methods, including hot mixing and cold processing, with or without the presence of virgin aggregate and/or virgin asphalt cement and/or rejuvenating agents and/or other suitable softening agents. The positive results of experiments to be described hereinafter in softening salvaged asphalts, for improving compatibility, and for improving adhesion to aggregate for pavement recycling using sewage, sludge-derived oil indicate the expanded potential use and therefore market value for sewage sludge-derived oil.

(f) AT LEAST ONE MODE FOR CARRYING OUT THE INVENTION

This is described by the following examples.

Example 1

Aged asphalt cement extracted from discarded old pavement was softened with sewage sludge-derived oil and Bow River asphalt, (a soft virgin asphalt). As well, a hard commercial asphalt was softened with both sewage sludge-derived oil and Bow River asphalt. The penetrations and viscosities for these examples are shown in Tables 1 and 2 and graphically in Figures 1 and 2.

Figure 1 is a graphical summary of the penetration tests previously described showing the blend of recovered asphalt and sewage sludge-derived oil (+150°C) vs. penetration at 4°C and at 25°C.

Figure 2 is a graphical summary of the viscosity tests previously described showing the blend of recovered asphalt and sewage sludge-derived oil (+250°C) vs. viscosity at 100°C and at 135°C.

TABLE 1

Penetration Comparison Between Recovered Asphalt Cement and Petro Canada 85/100 Asphalt When Different Additives are Added

PENETRATION (dmm)

	4°C	25°C
RAC	7	30
RAC + 2.1% SDO	7	40
RAC + 6.0% SDO	7	50
RAC + 12.2% SDO	11	98
RAC + 22.0% Bow River	13	80
PC 85/100	8	96
PC 85/100 + 6.0% SDO	14	159
PC 85/100 + 14.0% Bow River	15	148
Bow River	>300	n/a
SDO	>400	n/a

RAC = Recovered Asphalt Cement
SDO = Sewage Sludge-Derived Oil
PC = Petro Canada 85/100 Asphalt
The SDO fraction used was SDO +150°C
The Bow River fraction used was Bow River +454°C
n/a = Not analyzed

TABLE 2

Viscosity Comparison Between Recovered Asphalt Cement and Petro Canada 85/100 Asphalt When Different Additives are Added

KINEMATIC VISCOSITY (cSt)

	100°C	135°C
RAC	11039	866
RAC + 2.1% SDO	6883	719
RAC + 6.0% SDO	4794	624
RAC + 12.2% SDO	4807	413
RAC + 22.0% Bow River	3079	287
PC 85/100	n/a	332
PC 85/100 + 6.0% SDO	1690	218
PC 85/100 + 14.0% Bow River	1518	203
Bow River	67.34	21.44
SDO	21.95	7.25

RAC = Recovered Asphalt Cement
SDO = Sewage Sludge-Derived Oil
PC = Petro Canada 85/100 Asphalt
n/a = Not analyzed
The SDO fraction used was SDO +150°C
The Bow River fraction used was Bow River +454°C

A brief synopsis of the compositions (in weight %) of the virgin asphalts used, as carried out by the Asphalt Separation Test by ASTM D 4124, is as follows.

Asphalt	Asphaltene	Saturates	Nap/Arom.	Pol/Ar.
PC 85/100	16.1	14.3	34.2	35.3
PC 150/200	9.3	16.0	35.9	38.8

PC = Petro Canada Asphalts. Both asphalts are commercial asphalts graded according to penetration grades, i.e. 85/100 = penetration between 85 and 100 dmm.
Nap/Arom. = Naphthene Aromatics
Pol/Ar. = Polar Aromatics

The results of the tests performed on the commercial virgin asphalts used are summarized below:

Test	Units	PC 85/100	PC 150/200
Strp Imm.	%	36	20
Pen. 4,25,30°C	dmm	8,96,158	15,174,218
Flash Pt. COC	°C	324	316
Viscosity,60°C	Poises	1573	476.1
,135°C	cSt	332.1	191.8
Ductility,25°C	cm	+150	130
TriClEth.Sol'ty	%	99.93	99.95
TFOT Wt Loss	%	0.024	0
Pen.25°C	dmm	53	99
Ret.Pen.	%	53	99
Vis.60°C	Poises	3178	799.5
135°C	cSt	446.2	244
Ductility,25°C	cm	150+	n/a
Strp.Imm.	%	25	n/a

Strp Imm. = Stripping By Static Immersion
Pen. = Penetration
TriClEth.Sol'ty = Solubility in Trichloroethylene
TFOT = Thin-Film Oven Test
Ret.Pen. = Retained Penetration
Vis. = Viscosity

The results of Tables 1 and 2 indicate that small amounts of sewage sludge-derived oil soften hard asphalt and recycled asphalt as much as larger amounts of soft virgin asphalt. In particular, aged asphalt of 30 dmm penetration at 25°C is converted to an 85/100 asphalt by the addition of 12.2% sludge-derived oil; commercial 85/100 asphalt is converted to soft asphalt (150/200) by the addition of 6% sludge-derived oil. As well, the recycled asphalt blended with 12.2% sludge derived oil passes Ministry of Transport of Ontario specification for kinematic viscosity (minimum of 280 cSt) for 85/100 asphalt [test ASTM 2170-85]. The performance of sewage sludge-derived oil for softening hard asphalt and recycled asphalt at least matches the performance of Bow River asphalt but at significantly reduced amounts. If more Bow River had been added to the old asphalt to bring it to a specified penetration of 85/100 asphalt, then the viscosity would have been below the specified 280 cSt (minimum).

Example 2

The addition of sewage sludge-derived oil to recycled asphalt cement has been found to improve ductility.

In this example, 5% of +150°C sewage sludge-derived oil and 5% of +250°C sewage sludge-derived oil were added, respectively, to aged asphalt.

A comparison of the sewage sludge-derived oil containing more lighter and less lighter material as additives for aged asphalt is shown in Table 3.

TABLE 3

INCREASE OF DUCTILITY BY ADDITION OF SDO

	RAC V	5% SDO* in RAC V	5% SDO** in RAC V
Penetration @ 25°C (mm/10)	38	59	55
Viscosity @60°C (Poises)	11425	2896	3531
Ductility @25°C (cm)	31.5	137.0	101.2

* Sewage Sludge-Derived Oil+150°C was used

** Sewage Sludge-Derived Oil+250°C was used

SDO = Sewage Sludge-Derived Oil
RAC = Recovered Asphalt Cement

As shown in Table 3, above, the penetrations increased from 38 to 59 and 38 to 55 and the viscosities at 60°C were lowered from 11425 to 2896 and 3531 poises respectively. Figure 3 is a graphical summary of the penetration tests previously described showing the penetration vs. +350°C sewage sludge-derived oil additive blend with aged asphalt.

This shows that the material with lighter components is somewhat more effective for softening. Further, the sewage sludge-derived oil is quite effective in increasing ductilities from 31.5 to 137 and 101 cm respectively. ASTM D-946 specifies a minimum of 100 cm ductility for hard (85/100) asphalt cement. The increase in ductility demonstrates the compatibility of sewage sludge-derived oil with heavy asphaltenic materials.

<u>EXAMPLE 3</u>

Blends of sewage sludge-derived oil with aged asphalt when subjected to Thin-Film Oven Tests yield satisfactory results. This is shown below in Table 4 when a blend containing 9% of sewage sludge-derived oil in aged asphalt was subjected to the Thin-Film Oven Test.

TABLE 4

EFFECT OF OXIDATION

<u>Thin-Film Oven Test</u>

Asphalt	PEN25°C (Before)	TFOT Wt Loss %	PEN25°C (After)	Retained PEN %
0 % SDO, COMMIII 150/200	174	0	99	56.9
10 % SDO +250°C COMMIII 150/200	342	1.06	187	54.7
0 % SDO, RAC	32	0.028		
8.92 % SDO +250°C RAC	65	0.60	39	60

PEN = Penetration (dmm)
COMMIII = A commercial Asphalt
SDO = Sewage Sludge-Derived Oil
RAC = Recovered Asphalt Cement
TFOT = Thin-Film Oven Test

As also noted above, the retained penetration was 60% which passes the Ministry of Transport of Ontario and ASTM D-946 specifications.

EXAMPLE 4

The change in viscosities after Thin-Film Oven Tests is shown in Table 5.

TABLE 5

RECOVERED ASPHALT CEMENT BLENDS
VS. VISCOSITY AT 60°C

Asphalt Cement Blend	Viscosity @ 60°C (P)
RAC +0% SDO +250°C	29 720
TFOT Residue	45 210
RAC +9% SDO +250°C	5 330
TFOT Residue	9 583

RAC - Recovered Asphalt Cement
TFOT - Thin-Film Oven Test
SDO - Sewage-Sludge Derived Oil

This viscosity change is within the specifications required by those Canadian transportation agencies who set this specification.

With 85/100 and 150/200 penetration grade asphalts it has been found that at least 5% of the +150°C sewage sludge-derived oil can be added before the blends fail the Thin-Film Oven Test due to volatization of the lighter components. With the heavier aged asphalts considerably more can be added of the +150°C sewage sludge-derived oil; alternatively a higher initial boiling point sewage sludge-derived oil could be used. Those asphalts could be diluted with other materials, e.g. soft virgin asphalt.

EXAMPLE 5

The compatibility of sewage sludge-derived oil with heavy residual materials was then assessed. Sewage sludge-derived oil was added to CANMET hydrocracking pitch (S.P.111°C) and to ROSE$_{TM}$ residue (S.P.158°C) in equal proportions, heated until liquid and thoroughly mixed. An aged Athabascan asphaltene fraction was similarly treated with twice its weight of sewage sludge-derived oil.

A description of the characteristics of the above-referred-to CANMET hydrocracking pitch is as follows: Very heavy material. Typically 50 - 80% Asphaltenes.

Asphaltene content varies with the feedstock and the conversion rate selected for the process. (See U.S. Patent No. 4,683,005 July 28, 1987 M. A. Poirier).

A description of the characteristics of the above-referred-to ROSE$_{TM}$ residue is as follows:

Residuum Oil Supercritical Extraction (ROSE$_{TM}$) process by the phenomena of reverse solubility separates asphaltenes from heavy oil residues using propane or butane solvent. The characteristics of the hydrocracking pitch will vary according to the feed stock used, but typically very high in asphaltenes:

R & B softening Pt. 80-100°C

Viscosities	200-400 cSt @ 200°C
Nickel & Vanadium	100-300 wppm (high)

In the case of the hydrocracking pitch and the ROSE$_{TM}$ residue blends, the materials appeared to dissolve completely in the sewage sludge-derived oil. In the case of the asphaltenes, there appeared to be only a very small portion of skin-like material that did not dissolve in the sewage sludge-derived oil. This small amount of insoluble material probably was due to oxidation of the asphaltene fraction on ageing.

The mixtures were very viscous, on cooling, but no phase separation could be detected, even after several weeks. Thus, it is concluded that the sewage sludge-derived oil has an affinity for heavy asphaltic materials. The fact that the addition of sewage sludge-derived oil to aged asphalt markedly increases the ductilities as was shown above in Table 3 supports this conclusion.

The sewage sludge-derived oils are fairly immiscible with hydrocarbon fraction that have high saturated hydrocarbon contents. While it is not desired to be restricted by any theory, it is believed that there is a possibility that asphalts that are high in saturated hydrocarbons, in particular waxy components, may be incompatible when very large amounts of sewage sludge-derived oil are added. However, such asphalts would not be generally desired for paving.

EXAMPLE 6

The addition of sewage sludge-derived oil to aged asphalt can markedly improve the resistance to stripping of the rejuvenated asphalt from aggregates. This is shown in the bar graph shown in Figure 4, which shows the results of the stripping by static immersion test.

As seen in Figure 4, 9% of sewage sludge-derived oil was added to the aged asphalt. In addition to rejuvenation, this retained surface coverage after the stripping test was 100% for this rejuvenated asphalt and was only 42% for the aged asphalt. The result for the recycled asphalt blended with sludge-derived oil exceeds the Ministry of Transport of Ontario requirement of 95% coverage for hard asphalts.

CLAIMS

1. An asphaltic composition consisting essentially of: comminuted aged asphaltic pavement material; an effective amount, from 2% to 15% by weight, of a blend of an agent which is selected from the group consisting of a soft asphalt cement, a conventional asphalt cement, and a cutback asphalt, with a nitrogen-containing, adhesion-improving, anti-stripping agent comprising a sewage sludge-derived oil comprising a mixture of saturated aliphatic hydrocarbons, monoaromatic hydrocarbons, diaromatic hydrocarbons, polyaromatic hydrocarbons, polar compounds and basic, pyridine-soluble compounds, having the following elemental chemical composition: nitrogen, 3.42% to 5% by weight; oxygen, 5.8% to 6.9% by weight, sulphur, 0.3% to 0.8% by weight; hydrogen, 9.7% to 10.4%, and carbon, 76.9% to 79.8%.

2. The asphaltic composition of claim 1 wherein the amount of said sewage sludge-derived oil blend is from 2% to 12% by weight.

3. The asphaltic composition of claim 1 or claim 2, which is blended with soft virgin asphalt.

4. The asphaltic composition of claim 1, claim 2 or claim 3, and also including at least one additional agent which is selected from the group consisting of other asphaltic materials, at least one rejuvenating agent and at least one softening agent.

5. The asphaltic composition of any one of claims 1 to 4, inclusive, wherein said sewage sludge-derived oil has the following elemental composition: nitrogen, 3.42% to 4.95% by weight; oxygen, 5.84% to 6.89% by weight, sulphur, 0.34% to 0.83% by weight; hydrogen, 9.70% to 10.44%, and carbon, 76.92% to 79.76%.

6. A method for rejuvenation of salvaged, old asphaltic pavement material which comprises: incorporating, into said old asphaltic pavement, a sewage sludge-derived oil, or fraction thereof, said sewage sludge-derived oil comprising a mixture of saturated aliphatic hydrocarbons, monoaromatic hydrocarbons, diaromatic hydrocarbons, polyaromatic hydrocarbons, polar compounds and basic, pyridine-soluble compounds, having the following elemental chemical composition: nitrogen, 3.42% to 5% by weight; oxygen, 5.8% to 6.9% by weight, sulphur, 0.3% to 0.8% by weight; hydrogen, 9.7% to 10.4%, and carbon, 76.9% to 79.8%.

7. A method for improving the compatibility of aged asphalt in salvaged old asphalt paving materials with other asphaltic materials, which comprises incorporating, into

said old asphaltic pavement, with or without other asphaltic materials, and/or rejuvenating agents and/or suitable softening agents, a sewage sludge-derived oil, or a fraction thereof, said sewage sludge-derived oil comprising a mixture of saturated aliphatic hydrocarbons, monoaromatic hydrocarbons, diaromatic hydrocarbons, polyaromatic hydrocarbons, polar compounds and basic, pyridine-soluble compounds, having the following elemental chemical composition: nitrogen, 3.42% to 5% by weight; oxygen, 5.8% to 6.9% by weight, sulphur, 0.3% to 0.8% by weight; hydrogen, 9.7% to 10.4%, and carbon, 76.9% to 79.8%.

8. A method for improving the resistance to stripping of rejuvenated asphalt cement aggregate comprising the utilization of a sewage sludge-derived oil, or a fraction thereof, said sewage sludge-derived oil comprising a mixture of saturated aliphatic hydrocarbons, monoaromatic hydrocarbons, diaromatic hydrocarbons, polyaromatic hydrocarbons, polar compounds and basic, pyridine-soluble compounds, having the following elemental chemical composition: nitrogen, 3.42% to 5% by weight; oxygen, 5.8% to 6.9% by weight, sulphur, 0.3% to 0.8% by weight; hydrogen, 9.7% to 10.4%, and carbon, 76.9% to 79.8%.

9. The method of claim 6, claim 7 or claim 8, wherein said sewage sludge-derived oil has the following elemental composition: nitrogen, 3.42% to 4.95% by weight; oxygen, 5.84% to 6.89% by weight, sulphur, 0.34% to 0.83% by weight; hydrogen, 9.70% to 10.44%, and carbon, 76.92% to 79.76%.

10. The method of any one of claims 6 to 9, inclusive, wherein the amount of said sewage-derived oil is from 2 to 15% by weight.

PENETRATION WITH SDO CONTENT IN RECOVERED ASPHALT CEMENT

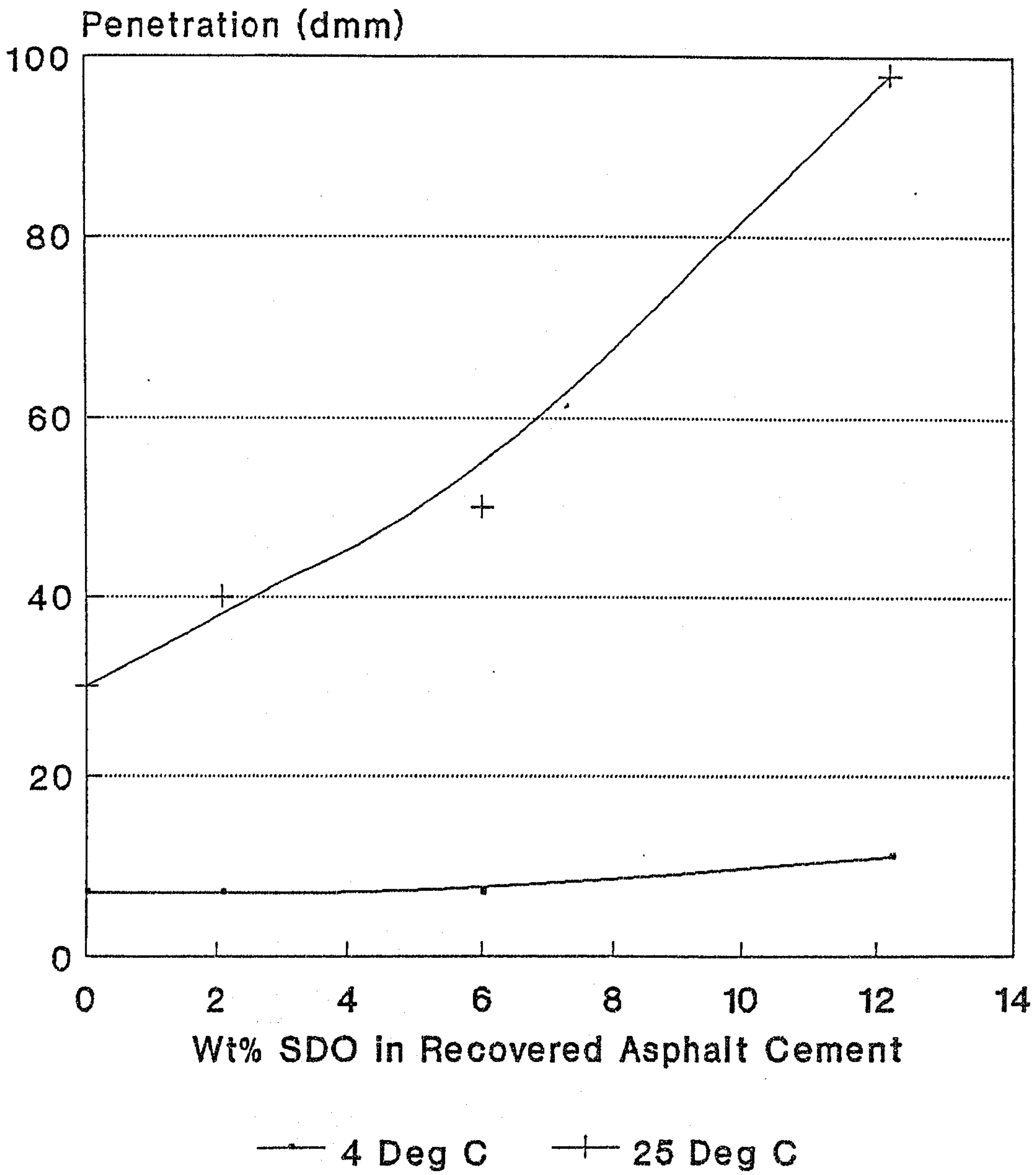

FIG. 1

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VISCOSITY WITH SDO CONTENT
IN RECOVERED ASPHALT CEMENT

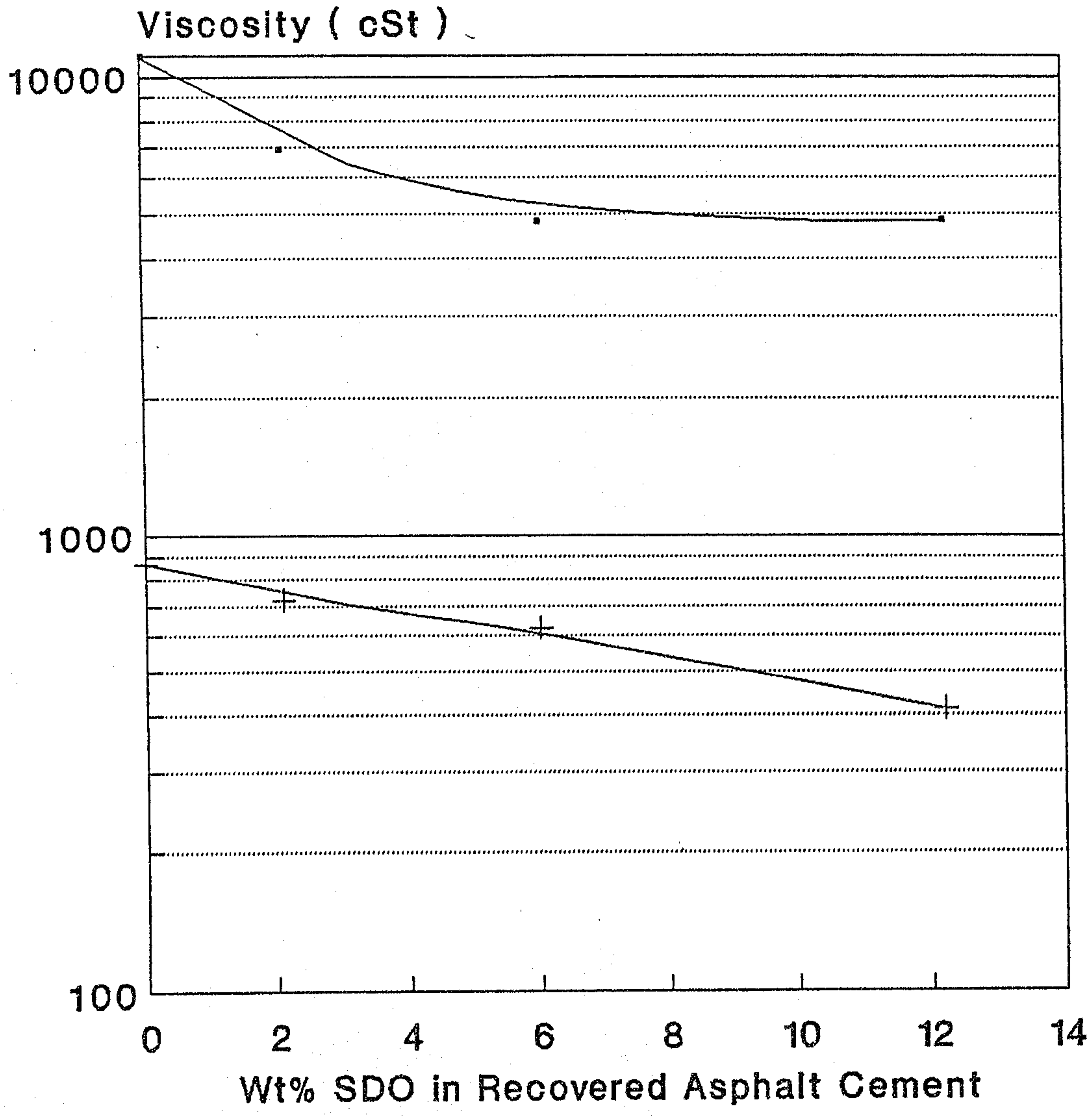


FIG. 2

McFadden, Fincham, Marcus & Allen

PENETRATION WITH SDO CONTENT
IN RECOVERED ASPHALT CEMENT

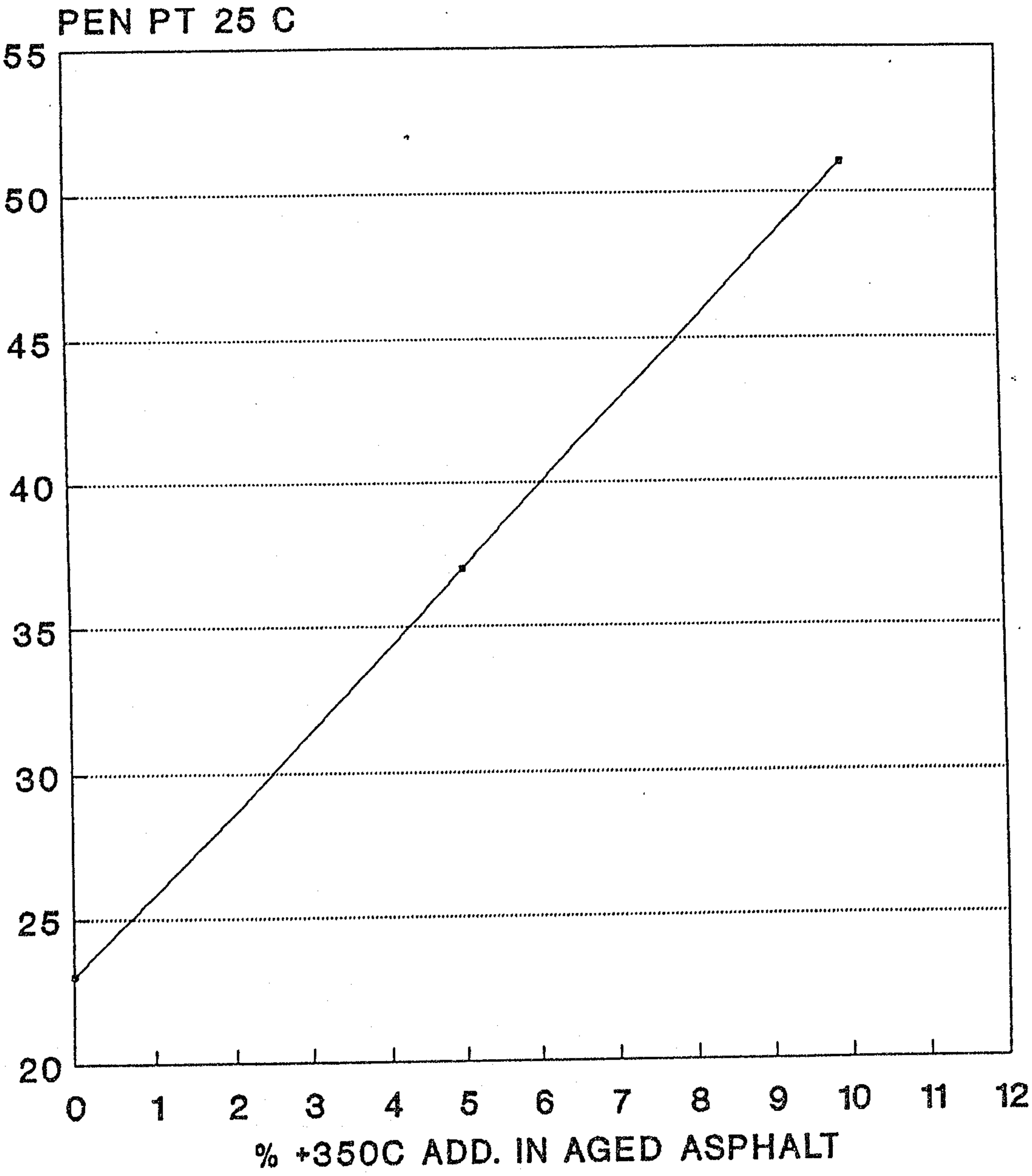
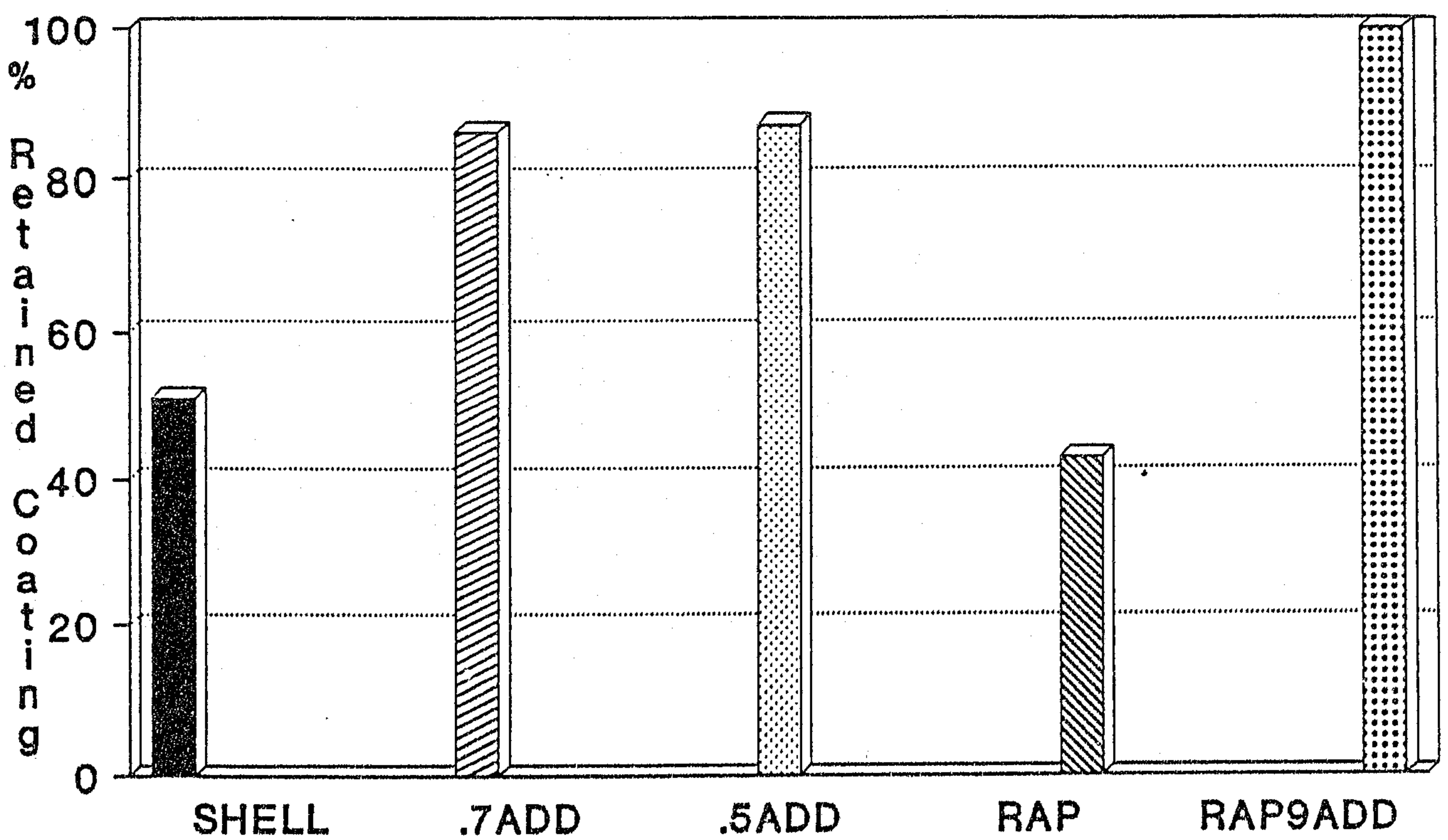


FIG. 3

STRIPPING BY STATIC IMMERSION
BLENDS WITH SHELL 85/100 ASPHALT
ADDITIVE +150C



AGGREGATE: JAIMESON

FIG. 4