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Study of Aging Characteristics of Recycled Asphalt Binders: Focusing on the Chemical Properties

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Abstract: After several years of service, the aromatic components in asphalt cement turn into asphaltenes, which result in stiffer, fragile and distressed pavements. In hot in-plant recycling operations, new binder (or recycling agent) and aggregate are mixed with reclaimed asphalt pavement (RAP) to minimize the negative effects of age hardening. As described in the literature, recycling agents should be highly aromatic to produce recycled pavement superior to original pavement. Many studies have been done studying physical properties and pavement performance of recycled asphalt mix, but rarely have they focused on the chemical properties. This study attempted to investigate the chemical components of recycled asphalt and their effects on recycled asphalt binder properties. The effectiveness of using different recycling agents in the recycled asphalt mixes was also evaluated. The results indicated that conventional AC-10 could be effective in softening severely aged RAP to produce asphalt binder with viscosity in the range of paving asphalt. However, recycled asphalt mixes with AC-10 as a rejuvenator show not only higher asphaltenes but also higher polar aromatics and lower naphthene aromatics. The two recycling agents, RA75 and RA250, however, showed beneficial effects in terms of both viscosity and chemical components. An asphalt binder aging index, Corbett Aging Index, based on calculations of asphaltenes and naphthene aromatics, is proposed as an effective parameter to sensitively detect aging severity of different recycled mixes.

***Key words**: Chemical components; Corbett method; Recycled hot-mix asphalt; Viscosity.*

Introduction

Hot-mix recycling of asphalt pavements has been used as a viable pavement rehabilitation method in Taiwan since 1998. Many kilometers of roads have used asphalt containing reclaimed asphalt pavement (RAP). Past experiences indicated that, in Taiwan, asphalt pavement generally require some types of rehabilitation works every four to eight years. Therefore, many asphalt pavements containing RAPs had undergone maintenance and/or repair works. Therefore, it is necessary to study the properties of the recycled asphalt pavement in order to understand the how these recycled pavements would behave after subjecting to another cycle of recycling.

Previous studies on recycled asphalt mixes have been mainly focused on physical properties and pavement performance; seldom has research been done in depth on their chemical properties. The objectives of this study are to investigate the chemical components of recycled asphalt and their influences in properties of recycled asphalt. The effectiveness of using various recycling agents in recycled asphalt pavements was also evaluated.

Literature Review

Majority of paving asphalt cement used today is obtained from processing crude oil. The compositions of asphalt are complex and are different from source to source. Asphalts are recognized as complicated colloidal systems of hydrocarbon material that, from a chemical point of view, are largely unknown [1]. Although the composition tests based on fractional separation do not consistently correlate with field performance, some useful information on asphalt chemistry has been observed as follows [2]:

1. Asphalt consists of significant heteroatom contents, which include nitrogen, oxygen, sulfur, vanadium, nickel, and iron.
2. The components of heteroatoms play an important role in determining physical properties of asphalt cement. The polar heteroatom compounds (functional groups) are capable of inducing intermolecular associations affecting physical properties such as boiling points, solubility, and viscosity. These polar compounds tend to be concentrated in the asphalt fraction of crude oil.
3. The molecular weight of asphalt compounds ranges from about 300 to 2,000. Yet, as a result of molecular associations, asphalt behaves as if it has a much higher molecular weight.
4. Aging of asphalt is associated with oxidation; an increase in the polar fractions upon aging results in, among other things, increased asphalt viscosity.
5. The composition, rheology, and durability of asphalt are unique to the crude blend from which the asphalt is refined.

Physical properties of asphalt are determined by its chemical compositions. The four chemical components of asphalt are asphaltenes, polar aromatic, naphthene-aromatics, and saturates. According to Corbett [3], the asphaltenes function as solution thickeners while fluidity is imparted by the saturate and naphthene aromatic fractions, which plasticize the solid polar aromatic and asphaltene fractions. The polar aromatic fraction imparts ductility to the asphalt. The complex flow properties of the asphalt are influenced by the combined actions of saturates and naphthene-aromatics. During the asphalt aging process, saturates remain the same while the solubilizing aromatics decrease in quantity. The

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increase in asphaltenes is due to the reaction between aromatics and oxygen. The increase in asphaltenes and decrease in solubilizing aromatics result in incompatibility because saturates and asphaltenes are not soluble in each other. This incompatibility causes a striking increase in viscosities and decreases in ductility [4].

After several years of service, asphalt aged due to oxidation. Binders recovered from aged asphalt pavement are much stiffer than desired. To reuse the aged asphalt pavements, in hot in-plant recycling operations, new binders and aggregate are mixed with RAP to produce asphalt mixes with desired properties. Recycling agents are often used in hot-mixed asphalt recycling operations. Mechanical mixing, diffusion, and compatibility influence the mixing of the old and new binder (i.e., solubility parameter and molecular weight distributions). Using a stage extraction method, in which inner and outer layers of the recycled binder film were extracted separately, the concept of the diffusion process was physically verified [5]. In a laboratory study that investigated the mixing of new and old binders during recycling operations, a microstructure model was used to illustrate the concept of asphalt being a continuous phase of relatively non-associated molecules in which associated molecules (asphaltenes) were dispersed. Asphaltenes are main viscosity-building components. Therefore, oxidative aging leads to an increase in asphalt viscosity by converting some maltenes into asphaltenes, and the viscosity of the remaining maltenes does not change significantly [6, 7]. The maltenes may then give a soft medium for diffusion.

Taiwan highway agencies began promoting hot mix recycling in 1998 [8]. Limiting the RAP content to 40%, conventional AC-10 asphalt binders were generally used in the recycling process. It further required that all hot asphalt mix plants processing HMA recycling be certified. As of 2006, more than 100 hot mix plants had been certified representing about to 61% of all hot mix plants in Taiwan.

Considering the fact that asphalt pavement rehabilitations were required once every four to eight years in Taiwan, many of the pavements containing RAP had undergone some types of maintenance and/or repair works. To achieve adequate pavement performance, properties of recycled asphalt pavement after aging needed to be understood. What binder properties would be important in affecting the performance of pavements constructed with RAP? Is there a need to use recycling agent in Taiwan? Viscosity alone may no longer be a reliable determining factor. Study of properties of chemical components of asphalt binders along with field performance should be taken into account in order to guarantee the sustainability of recycled pavement.

Laboratory Study Procedures

This study consisted of two major components. The objective of the first part was to evaluate the effectiveness of using conventional AC-10 asphalt cement as a rejuvenator in producing recycled asphalt mixtures and the second studied effects of three recycling agents, AC-10 asphalt cement, RA75, and RA250, on recycled asphalt mixtures.

In the first phase of the study, three RAPs with different aging severity, designated as high (H), medium (M) and low (L), from different hot mix plants were used. The evaluation consisted of the following steps:

- Conducting Marshal mix design to establish a reference proportion using virgin materials (setting up target values of asphalt content and aggregate gradation for recycled mixes).
- Measuring viscosity and chemical components of the AC-10.
- Mixing three different amounts of each RAP with new AC-10 binder and virgin aggregate, producing total of nine recycled asphalt mixes. Amounts of RAPs used were 60, 40, and 20% by weight of total binders in the recycled mixes. The nine mixes were designated by the aging severity and percentage of RAPs. For instances, M40 represented a recycled asphalt containing 40% RAPs of medium aging severity.
- Recovering asphalt binders from the nine recycled asphalt mixes as well as the three RAPs using the Reflux Extraction (ASTM D2172 method B) and Rotorvapor Recovery methods [9].
- To further study the aging characteristics of asphalt mixes containing RAPs, three of the nine recycled mixes, H60, M40, and L20, were put into a forced draft oven at 85˚C [10] for three different durations, 4, 8, and 12 days, and the binders from the three oven-aged recycled asphalt mixes were recovered following procedure used previously.
- Separating ashes from the recovered asphalt binder solution by centrifuging as specified in ASTM D1856.
- Measuring viscosity at 60˚C and chemical components of the recovered binders. The four chemical components, asphaltenes (A), polar aromatics (PA), naphthalene aromatics (NA), and saturates (S), were determined using the selective adsorption-desorption method (ASTM D4124 Method B/Corbett Method [11]).

The purpose of second part of the project was to investigate the effects of three rejuvenators, conventional AC-10 asphalt cement, and ASTM classified Recycling Agents RA75 and RA250 [12], on the properties of the recovered binders from asphalt mixes containing RAPs. The investigation consisted of the following:

- Measuring chemical components of the three new binders and three recovered binders from the three RAPs with high, medium and low severity of aging.
- Mixing the three new binders with the oven-aged and field-obtained RAPs with the highest degree of aging (40% of RAP), producing six recycled asphalt mixes.
- Recovering binders from the six recycled asphalt mixes and measuring viscosity at 60˚C and chemical components of the recovered binders.
- The three mixes containing 40% oven-aged RAPs were subjected to additional aging in the same forced draft oven at 85˚C for two durations, 4 and 8 days. Viscosities and chemical components were then measured on the recovered binders. Three parameters were used to detect aging severity of asphalt mixes.

Test Results and Data Analysis

According to the research plan, Table 1 lists the measured viscosities and Corbett components of the binders recovered from the nine different recycled asphalt mixes as well as the three RAPs.

Table 1. Viscosities and Corbett Components of Different Blends of Recycled Mix.

Type of recover binder	New binder ratio[1]	Viscosity at 60°C (poises)	Corbett Components (%)			
			A	PA	NA	S
H100	0	194,789	27.5	54.1	10.4	7.9
H60	0.50	10,560	20.2	44.3	26.4	9.0
H40	0.67	4,638	19.1	42.9	29.0	9.1
H20	0.83	2,286	17.1	42.7	31.2	9.0
M100	0	95,994	25.4	46.8	19.8	8.1
M60	0.56	6,202	19.0	45.3	27.1	8.6
M40	0.71	3,351	18.2	43.4	29.4	9.0
M20	0.85	1,962	16.2	43.8	31.2	8.8
L100	0	12,559	19.9	44.3	27.2	8.1
L60	0.49	3,567	17.3	34.1	41.0	7.6
L40	0.66	2,400	16.4	30.4	43.5	9.8
L20	0.83	1,646	15.7	29.6	45.9	8.8

Note:
[1] Calculated by using asphalt content of RAP, % of RAP used, and total binder content.

The three RAPs were designated as H100, M100, and L100. With known properties and amount used of the AC-10 and the RAPs in the mixes, viscosities of recovered binders from the nine asphalt mixes were estimated using the procedures described in the ASTM D4887 [13]. A comparison between the estimated and measured viscosities of the nine recovered binders is shown in Fig. 1. As presented in this figure, a high correlation exists between the estimated and measured viscosities. During the hot mix recycling operations, the viscosity of the binder in the recycled asphalt mixes can be estimated from ASTM D4887 procedure and then adjusted using the regression equation to represent the measured viscosities.

Similarly, the chemical components of the nine recovered binders were calculated using Eq.(1) listed below and the estimated values were compared to the actual measurements from the recycled mix. High correlations were observed between measured and estimated values for asphaltenes and total aromatics, TA, (PA + NA). Figs. 2 and 3 show the linear correlation between calculated and measured asphaltenes contents and total aromatics with R-square values of

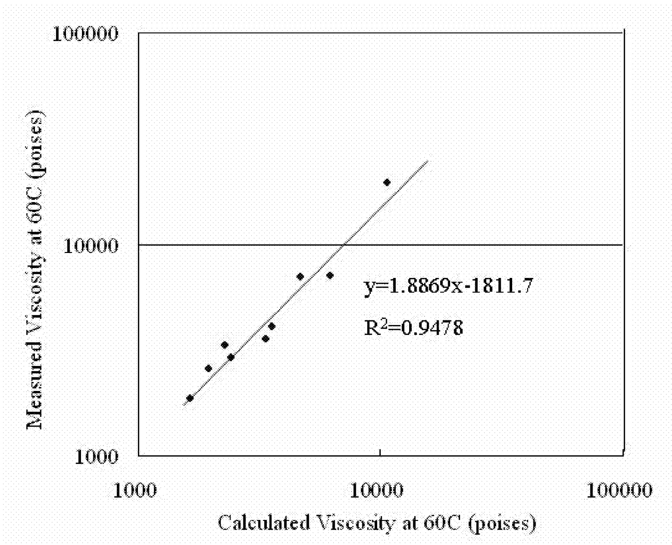


Fig. 1. Correlation of Measured and Calculated Viscosities of Recovered Binders from Different Recycled Mixes.

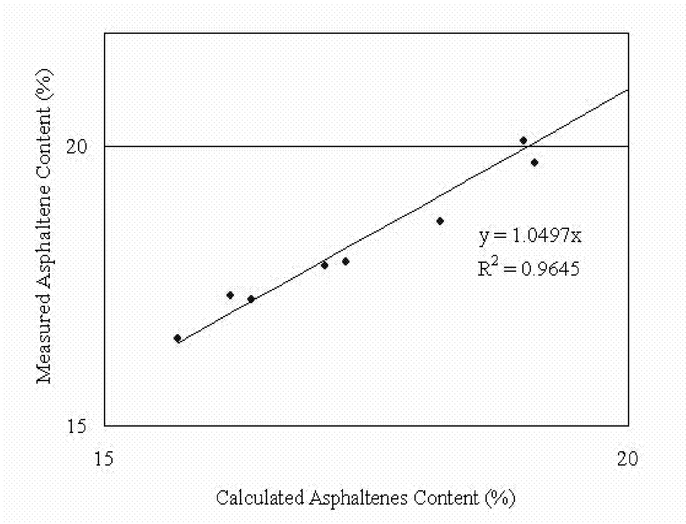


Fig. 2. Correlation of Measured and Calculated Asphaltenes Content of Recovered Binders from Different Recycled Mixes.

0.9645 and 0.8756, respectively. Again, these regression equations can be used to adjust chemical components estimated based on proportions of old and new materials used.

$$CC_{rm} = p_{ob} \times CC_{ob} + p_{nb} \times CC_{nb} \quad (1)$$

Where
CC_{rm} is chemical component of recycled mix (%),
CC_{ob} is chemical component of old binder (%),
CC_{nb} is chemical component of new binder (%),
p_{ob} is the fraction of old binder in the total binder of recycled mix,
p_{nb} is the fraction of new binder in the total binder of recycled mix.

As stated in the laboratory test procedures, three recycled mixes, H60, M40, and L20 were subjected to accelerated aging in the oven for different durations. Table 2 shows the viscosities and Corbett components of binders recovered from the three recycled mixes aged in the oven for different durations. Also shown in this table are

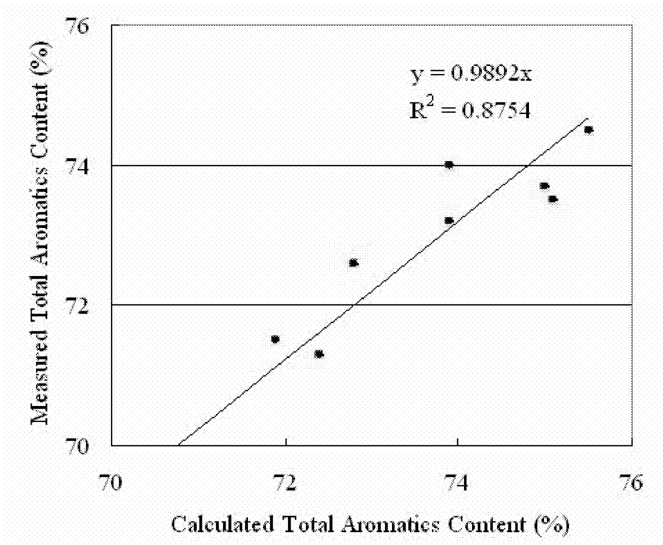


Fig. 3. Correlation of Measured and Calculated Total Aromatics Content of Recovered Binders from Different Recycled Mixes.

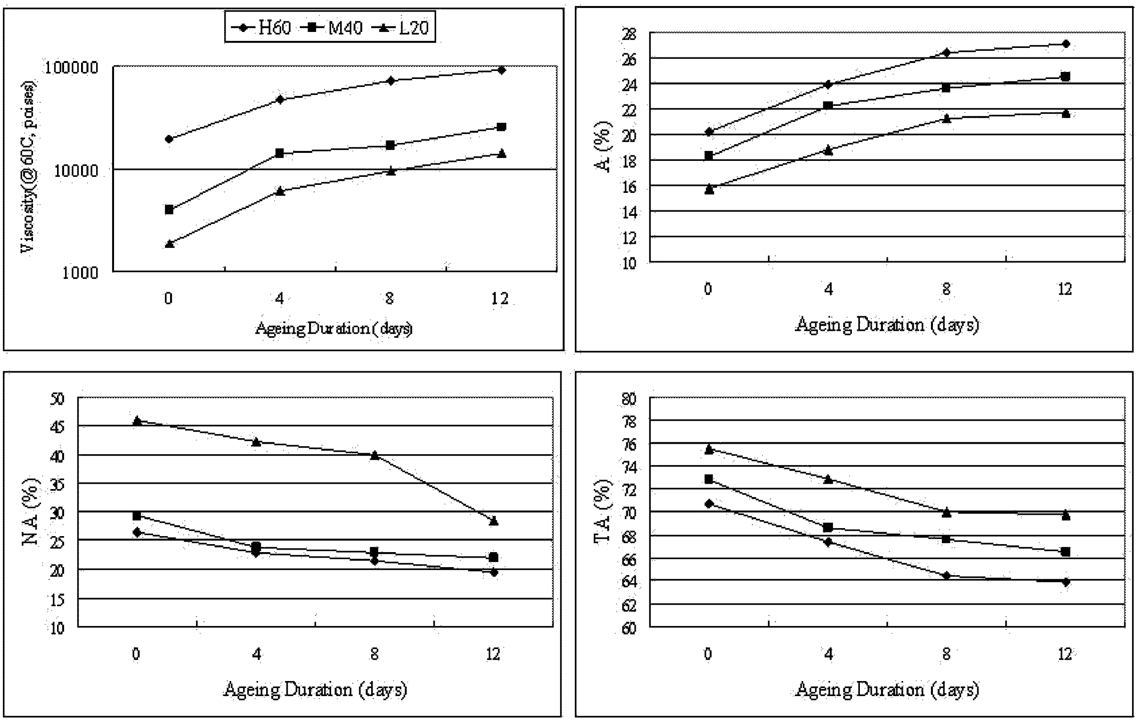


Fig. 4. Plots of Viscosity and Selected Components versus Oven Aging Durations Obtained from Recovered Binders of Different Recycled Mixes.

the total aromatics and the ratios of A/NA. To investigate the aging characteristics, viscosities and percent of A, NA, as well as TA of the recovered binders were plotted against oven aging durations, as shown in Fig. 4. One can see that asphaltenes and viscosity increase immensely while naphthene aromatics and the total aromatics decrease rapidly as the binders begin to age; the increasing and decreasing rates slow down as the binders grow older. The lowest total aromatics content is below 64%, similar to that of high aging severity RAP obtained from the field. Pavements with viscosity higher than 100,000 poises may still perform well but it is uncertain that a binder with such a low aromatic content could be recycled back into pavements and show adequate performance.

The objective of the second part of the study was to investigate the effects of three rejuvenators (binders), conventional AC-10 asphalt cement, and ASTM classified Recycling Agents RA75 and RA250, on the properties of the recovered binders from asphalt mixes containing RAPs. Measured chemical components of the six different asphalts binders (three different new binders and three recovered binders from the RAPs) are shown in Fig. 5. In Fig. 5, the *y*-axis shows the accumulated percentage of each chemical component, while the *x*-axis shows the different types of binders. As can been observed from this figure, as the binder ages (from AC-10 to LRAP, MRAP, and HRAP), asphaltenes increase, while naphthene aromatics decrease, and saturates remain the same. Even though the four binders on the left side of Fig. 5 were collected from different sources, the trends of change for chemical components due to aging are similar to those presented in other literatures. The two recycling agents, RA75 and RA250, show higher aromatics content and very low asphaltenes.

The softening effects of the three binders (AC-10, RA75, and RA250) are presented in Table 3. Recovered asphalt binders with the highest viscosities form both oven-aged and field obtained

Table 2. Viscosities and Corbett Components of the Three Recycled Mixes Aged in the Oven for Different Durations.

Type of Mix	Oven aging duration (days)	Viscosity at 60°C (poises)	Corbett Components (%) A	PA	NA	S	TA	A/NA
H60	0	19,573	20.2	44.3	26.4	9.0	70.7	0.765
	4	47,223	23.9	44.6	22.8	8.7	67.4	1.048
	8	72,818	26.4	43.0	21.4	9.2	64.4	1.234
	12	92,649	27.1	44.4	19.5	9.1	63.9	1.390
M40	0	3,950	18.2	43.4	29.4	9.0	72.8	0.619
	4	14,079	22.2	44.8	23.8	9.2	68.6	0.933
	8	16,995	23.6	44.8	22.8	8.8	67.6	1.035
	12	25,580	24.5	44.6	21.9	9.0	66.5	1.119
L20	0	1,888	15.7	29.6	45.9	8.8	75.5	0.342
	4	6,142	18.8	30.7	42.2	8.4	72.9	0.445
	8	9,510	21.2	30.1	39.9	8.8	70.0	0.531
	12	14,184	21.7	41.3	28.5	8.5	69.8	0.761

Table 3. Comparisons on the Softening Effect of New Binders on Oven and Field Aged RAPs.

Item		Oven aged			Field aged		
Viscosity of RAP (@60 °C, poises)		92,649			194,789		
New binder ratio[1]		0.60			0.67		
Type of new binder		AC-10	RA250	RA75	AC-10	RA250	RA75
Viscosity of recovered binder from recycled mix(@60 °C ,poises)		7,624	1,462	445	7,070	1,376	456
Corbett components of recycled mix (%)	A	19.3	11.6	10.9	19.1	11.5	9.6
	PA	43.3	30.9	26.0	42.9	27.6	25.3
	NA	27.7	45.1	47.1	29.0	48.8	50.4
	S	9.8	12.4	15.9	9.1	12.1	14.7

Note:
[1]Calculated by using asphalt content of RAP, % of RAP used, and total binder content.

samples were mixed with the three types of new binders to produce recycled asphalt mixes. 40% of RAPs was used on all recycled mixes. The three different binders show a similar softening effect on the two RAPs in terms of viscosities as well as chemical components alterations, shown in Table 3.

From the table, it is apparent that all three binders show excellent softening ability, as evidenced by the enormous reductions in viscosities of all six recycled asphalt mixes. However, it should be noted that although AC-10 could reduce the viscosity of severely aged RAP, comparing to recovered binders from other two recycled mixes containing RA75 and RA250, the AC-10 recycled mixes show not only higher asphaltenes but also higher polar aromatics and lower naphthene aromatics. It is uncertain that recycled asphalt mixes containing AC-10 as rejuvenators would perform adequately in the field, since higher contents of asphaltenes and polar aromatics would cause higher viscosity. The two recycling agents, on the other hand, could have beneficial effects to severely aged RAPs from both points of view of viscosity and chemical components.

The three recycled asphalt mixes containing field aged asphalt, as listed in Table 3, were put into a forced draft oven at 85°C for 4 and 8 days, and the binders were recovered and their viscosities and chemical components were determined. The test results are presented in Table 4. Also shown in this table are three parameters proposed for detecting aging conditions of recycled asphalt mixes.

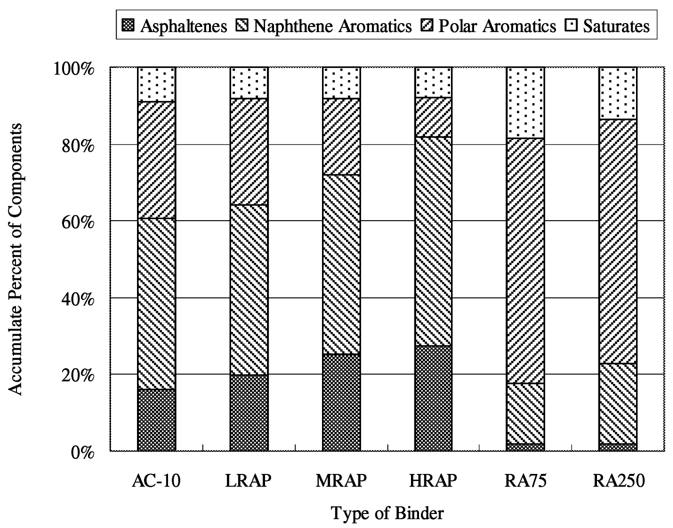


Fig. 5. Chemical Components in Accumulate Percent of Different Binders.

The three parameters were the conventional Aging Index (AI), the NA Ratio, and the Corbett Aging Index (Corbett AI), and are defined as follows:

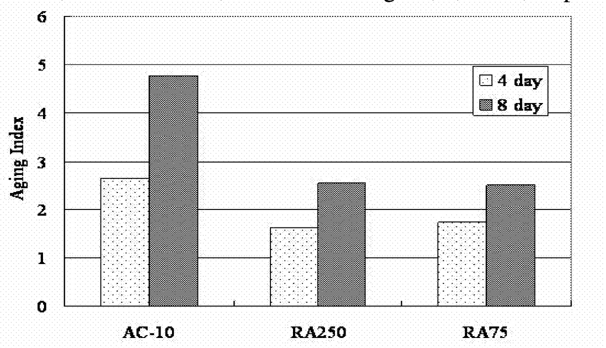
AI = viscosity of mixes before aging/viscosity of mixes after aging
NA Ratio = NA of mixes before aging/ NA of mixes after aging
Corbett AI = A/NA of mixes before aging/ A/NA of mixes after aging

The three parameters were evaluated to determine if they can be used in predicting the aging conditions of the recycled asphalt mixes. The following linear statistical model was used to perform a two-factor analysis of variance (ANOVA) without interaction to assess the effectiveness of the rejuvenators.

$$y_{ij} = \mu + \tau_i + \beta_j + \varepsilon_{ij} \qquad \begin{cases} i = 1, 2 \\ j = 1, 2, 3 \end{cases} \tag{2}$$

Where
y_{ij} is the aging index of the i^{th} aging duration and the j^{th} new binder,
μ is the overall mean,
τ_i is the effect of the two different aging severities,
β_j is the effect of the new binder, and
ε_{ij} is a random error component.

The results of the ANOVA on the three aging parameters, AI, NA Ratio, and Corbett AI, are shown in Figs. 6, 7, and 8, respectively.



ANOVA

Source	SS	DF	MS	F	P-value
Binder	2.435112	2	1.717556	6.031631	0.142215
Age	2.428248	1	2.428248	8.527405	0.099989
Error	0.569516	2	0.284758		
Total SS	6.432877	5			

Fig. 6. Plots of Aging Index along with the ANOVA for Differentiating Effects of Binders and Aging Durations

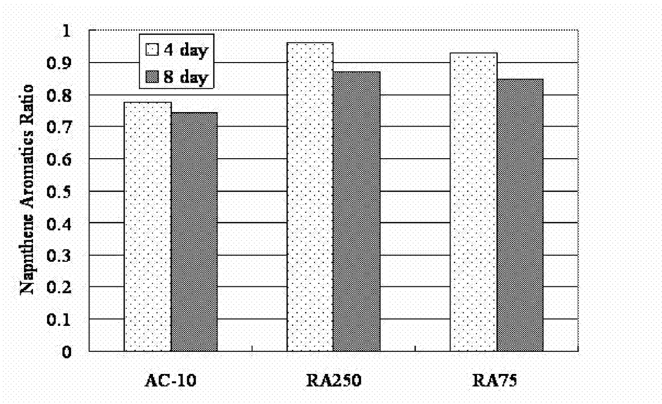
Table 4. Comparison on the Effect of Different New Binders on Oven Aging of Recycled Mixes Using Different Parameters.

Type of new binder	Oven AD[1] (days)	Viscosity at 60°C (poises)	AI	Corbett Components (%) A	PA	NA	S	NA ratio	A/NA	Corbett AI
AC-10	0	7,070	--[2]	19.1	42.9	29.0	9.1	--	0.659	--
	4	18,640	2.636	23.3	44.9	22.5	9.3	0.776	1.062	1.612
	8	33,751	4.774	24.6	45.5	21.6	8.3	0.745	1.139	1.728
RA250	0	1,376	--	11.5	27.6	48.8	12.1	--	0.236	--
	4	2,227	1.618	12.4	29.5	46.9	11.3	0.961	0.264	1.119
	8	3,501	2.544	13.3	32.3	42.4	12.0	0.869	0.314	1.330
RA75	0	456	--	9.6	25.3	50.4	14.7	--	0.190	--
	4	795	1.743	11.4	26.5	46.8	15.3	0.928	0.244	1.284
	8	1,138	2.496	12.6	30.6	42.8	14.0	0.849	0.294	1.547

Note:

1. Oven Aging duration in days.

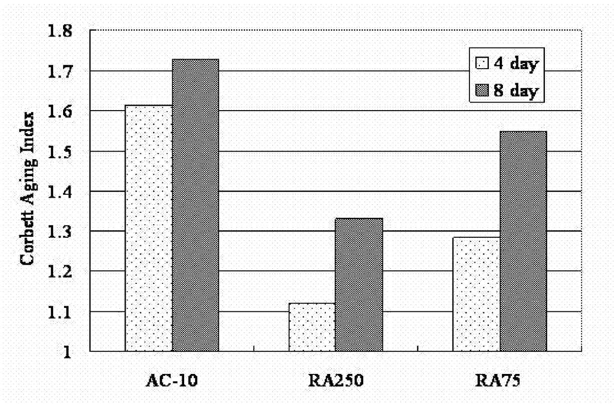
2. Not applicable.



ANOVA

Source	SS	DF	MS	F	P-value
Binder	0.027304	2	0.013652	26.44914	0.036431
Age	0.006801	1	0.006801	13.17533	0.068224
Error	0.001032	2	0.000516		
Total SS	0.035137	5			

Fig. 7. Plots of Naphthene Aromatics Ratio along with the ANOVA for Differentiating Effects of Binders and Aging Durations.



ANOVA

Source	SS	DF	MS	F	P-value
Binder	0.199814	2	0.099907	35.96155	0.027055
Age	0.058017	1	0.058017	20.88308	0.044699
Error	0.005556	2	0.002778		
Total SS	0.263387	5			

Fig. 8. Plots of Corbett Aging Index along with the ANOVA for Differentiating Effects of Binders and Aging Durations.

From these figures, it can be observed that the conventional AI is not an adequate indicator for detecting aging conditions of recycled asphalt mixes, as indicated by the relatively high P-values. The Corbett AI seems to be the best parameters for predicting the aging severity of recycled asphalt mixes. Therefore, the Corbett AI is proposed to be an indicator of aging conditions of asphalt mixes, and is defined as follow:

$$\text{Corbett Aging Index} = \frac{A_{aged}/NA_{aged}}{A_{unaged}/NA_{unaged}} \tag{3}$$

Where

A_{aged} and A_{unaged} are asphaltenes components in percent of aged and unaged binders, respectively, obtained by Corbett method.

NA_{aged} and NA_{unaged} are naphthene aromatics components in percent of aged and unaged binders, respectively, obtained by Corbett method.

Conclusions

By using the selective adsorption-desorption method, this study attempted to investigate the chemical components of recycled asphalt and their effects on recycled asphalt binder properties. The effectiveness of using different recycling agents in the recycled asphalt mixes was also evaluated. The following conclusions were derived from this study.

1. The asphaltene as well as the total aromatics content of recycled mix could be calculated with rational reliability by using the new binder ratio and the components of old and new binders.
2. Consistent with previous studies, saturates remain the same, asphaltenes increase and naphthene aromatics decrease with the aging asphalt binder.
3. From the point of view of viscosity, AC-10 could be effective in reducing the viscosity of severely aged RAP. However, the high asphaltenes and polar aromatics contents and low naphthene aromatics could have adverse effects on asphalt aging properties.
4. The two recycling agents evaluated in this study, RA75 and RA250, could soften severely aged RAP effectively both in terms of viscosity and chemical components.
5. The Corbett Aging Index, which is based on the calculations of asphaltenes and naphthene aromatics, is an effective parameter to sensitively detect aging severity of different recycled mixes.

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