Development of a Modified Rotavapor Apparatus and Method for Short-Term Aging of Modified Asphalts

OKAN SIRIN, CHUANG-TSAIR SHIH, MANG TIA, AND BYRON E. RUTH

An alternative laboratory asphalt aging process that could be used to simulate the aging effects of hot mixing on modified asphalts was developed and evaluated. The rotavapor apparatus, which has been used for recovery of asphalt from solution, was modified to work as an aging device for asphalts and modified asphalts. The rotavapor apparatus was modified such that the vacuum connection was replaced by an air pump with a controlled air flow. To reduce the variation of aging condition due to variation of the temperatures in the oil bath of the rotavapor apparatus, an insulated covering for the oil bath was constructed and used in this aging process. Evaluation results indicate that the aging severity of the modified rotavapor aging process is affected by variables such as process temperature, duration, and sample weight. All these factors could be adjusted to achieve the desirable level of asphalt aging. Because of the flexibility in controlling these variables in the modified rotavapor process, it appears that this method could be used to simulate the aging effects of hot mix plant process effectively.

In evaluating the potential performance of an asphalt binder in service, it is essential to characterize the properties of the asphalt binder after it has been subjected to the hot mixing process. At present, there are two standard processes that can be used to simulate the aging of an asphalt at a hot mix plant. They are the thin film oven test [(TFOT) ASTM D1754, AASHTO T179] and the rolling thin film oven test [(RTFOT) ASTM D2872, AASHTO T240]. The Strategic Highway Research Program (SHRP) Superpave binder specifications require that an asphalt binder to be evaluated is to be subjected to a standard RTFOT followed by a pressure aging vessel (PAV) process (which simulates several years of field aging).

Study by the University of Florida and the Florida Department of Transportation (FDOT) has shown that, although this SHRP-proposed aging method appears to be suitable for use on conventional asphalts, problems were encountered when modified asphalts were used. It was found that asphalts modified with crumb rubber and styrene butadiene rubber tended to spill out from the RTFOT bottles during the RTFOT process. When the TFOT process was used in stand of the RTFOT process, it was found that a thin skin tended to form on the surface of some modified asphalt samples, which reduced the homogeneity and aging of the samples (*1,2*). These problems with the RTFOT and TFOT processes on modified asphalts have also been reported by other researchers in the country (*3*). Therefore, there is a need to have a more suitable aging procedure for simulation of hot mixing on modified asphalts in place of the RTFOT and TFOT procedures. This paper presents the development of an alternative aging method using a rotavapor apparatus.

BACKGROUND ON THE ROTAVAPOR APPARATUS

The rotavapor apparatus is commonly used for the recovery of asphalts from solutions in the standard test method ASTM D5404. The rotavapor apparatus is presently used by FDOT for recovery of asphalt from solvents after asphalt extraction. The apparatus consists of a rotating distillation flask that is tilted at an angle and partially immersed in a heated oil bath at a controlled temperature. In the recovery test, the solution containing the asphalt to be recovered is placed in the rotating flask and subjected to a partial vacuum. The rotating flask is connected to a condenser tube and a receiver flask, which condenses and collects the solvent driven off from the rotating flask. A flow meter is used to control the rate of nitrogen or carbon dioxide blown into the flask.

The rotavapor apparatus is also used as an aging device in a German standard test (DIN 52016) for determining the mass loss and change of properties of asphalts due to heating. The rotavapor apparatus used is similar to that used in ASTM D5404 for recovery of asphalt, with the exception that the condenser tube and receiver flask are not used and the vacuum pump is replaced by an air pump. In this standard test, 100 g of asphalt is placed in the rotating flask, which is tilted at 45 degrees and submerged in a 165°C (329°F) oil bath, and rotated at 20 rpm. After the asphalt sample has been heated for 10 min in the rotating flask, air at ambient temperature is introduced into the flask at a rate of 500 mL/min, and the asphalt sample is aged in the rotating flask for an additional 150 mins. After this aging process, the mass loss of the asphalt is determined.

The rotavapor apparatus is a more versatile aging device than the thin film oven or rolling thin film oven. It can have the following advantages over these two conventional aging tests:

1. Different amounts of asphalt could be placed in the rotating flask for aging. The possible use of a larger sample could eliminate the need to combine two or more samples to get enough materials to run the SHRP PAV or other tests.
2. There is better and more consistent heat transfer between the asphalt and the heated oil in the rotavapor apparatus due to the greater thermal mass of the oil as compared with the air in an oven.
3. Different temperatures could be used easily.
4. Different flow rates of the injected air could be used easily.
5. Different sizes of rotating flask could be used.
6. Flask rotation prevents skin formation on the asphalt.
7. The size and tilting of the flask make spillage impossible.
8. Different rotating speeds could be used easily.

Department of Civil Engineering, University of Florida, Gainesville, FL 32611-6580.

PRELIMINARY EVALUATION OF THE ROTAVAPOR AGING APPARATUS

A rotavapor apparatus, which was originally used for recovery of asphalt from solvents after asphalt extraction, was modified to work as an aging device for asphalts and modified asphalts. The apparatus was modified such that the vacuum connection was replaced by an air pump with a controlled air flow. The schematic of the modified rotavapor aging apparatus is shown in Figure 1.

The rotavapor aging apparatus (as set up) was evaluated by running it on an AC-30 asphalt cement and a crumb rubber–(CR-) modified asphalt (AC-30 and 10 percent crumb rubber with a nominal size No. 80 sieve). The CR-modified asphalt was made in the laboratory by blending the AC-30 with the CR at 170°C using a high shear mixer. Two different sample sizes (50 and 75 g), two process temperatures (163 and 185°C), two process durations (60 and 85 min), one air flow rate (4000 mL/min), and one rotating speed (60 rpm) were used in the rotavapor aging process. The AC-30 asphalt and the modified asphalt were also aged by the standard TFOT and RTFOT for comparison purposes.

A penetration test at 25°C and a Brookfield viscosity test at 60°C were performed on the original binders and on the aged binders after these various aging processes. One sample was used for each combination of test conditions.

Figure 2 shows the comparison of the effects of the standard TFOT and RTFOT on the penetration of the AC-30 asphalt and the CR-modified asphalt with those of the rotavapor aging process (using 50-g samples and 85-min duration) at 163 and 185°C. The corresponding effects for Brookfield viscosity at 60°C are shown in Figure 3. It can be seen from Figures 2 and 3 that the aging severity of the rotavapor process at 163°C is slightly greater than that of the TFOT but slightly less than that of the RTFOT. The aging severity of the rotavapor process at 185°C is much greater than that of both the TFOT and RTFOT processes.

The effects of sample size, process duration, and process temperature on the rotavapor process were evaluated. Figures 4 and 5 show the effects of these factors on penetration at 25°C and Brookfield viscosity at 60°C, respectively, for the CR-modified asphalt aged by the rotavapor process. It can be clearly seen that sample size, process duration, and process temperature all have significant effects on aging severity. Smaller sample size, longer process duration, and higher process temperature produce significantly more severe aging effects.

MODIFIED ROTAVAPOR AGING APPARATUS

Preliminary experimentation with this rotavapor aging apparatus showed that the oil bath was a major source of variation within the procedure and needed to be improved. The oil bath was exposed directly to the ambient air in the laboratory. When the oil bath was in use, it was losing heat to the environment through the side wall of the container as well as through the surface of the oil. Temperature measurement at different locations in the oil bath had shown a great variation of temperature. This condition of large temperature variation could result in the following two major problems:

1. The asphalt binder in the rotating flask would not be heated evenly.
2. It would be extremely difficult to produce a consistent and repeatable aging effect on the asphalt binder in the rotating flask. The aging effect would be greatly influenced by factors such as (*a*) the height of the oil in the container, (*b*) the depth to which the rotating flask is placed within the oil bath, (*c*) the position of the thermal probe (used to control the heater) within the oil bath, and (*d*) the ambient air temperature.

To improve uniformity of temperature, the oil bath was modified by constructing a well-insulated box and covering the oil bath with it.

Since the maximum heating temperature of the oil bath is about 175°C, the insulated box was designed to sustain a temperature up to 200°C. The outside wall of the box was made of 24-gauge galvanized steel sheet; the inside wall was made of a cold-rolled steel. A 2.5-cm-thick insulating fiber material was placed between the galvanized steel sheet and the cold-rolled steel. In order to be able to place the flask into the oil bath easily, a 229 x 254-mm door with a 102x102-mm glass window was placed on the front side of the insulated box. The purpose of the glass window on the door is to enable

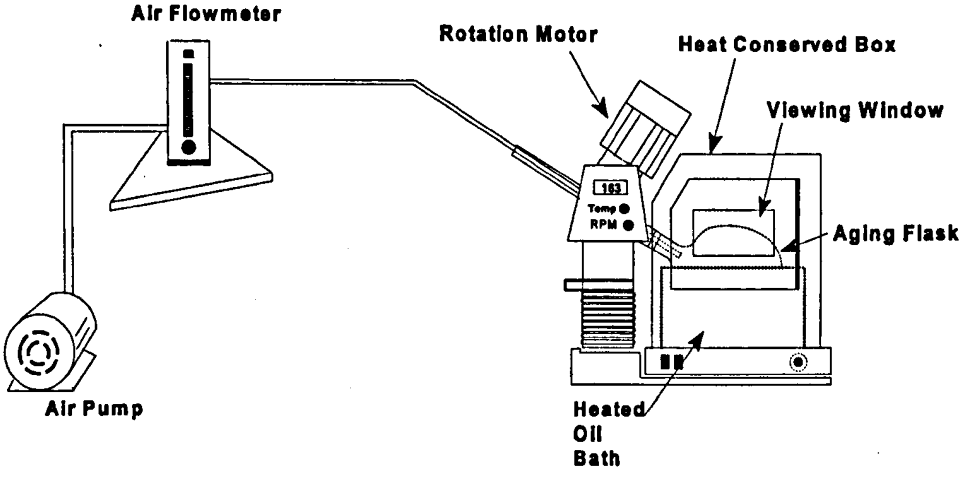

FIGURE 1 Schematic representation of modified rotavapor aging apparatus.

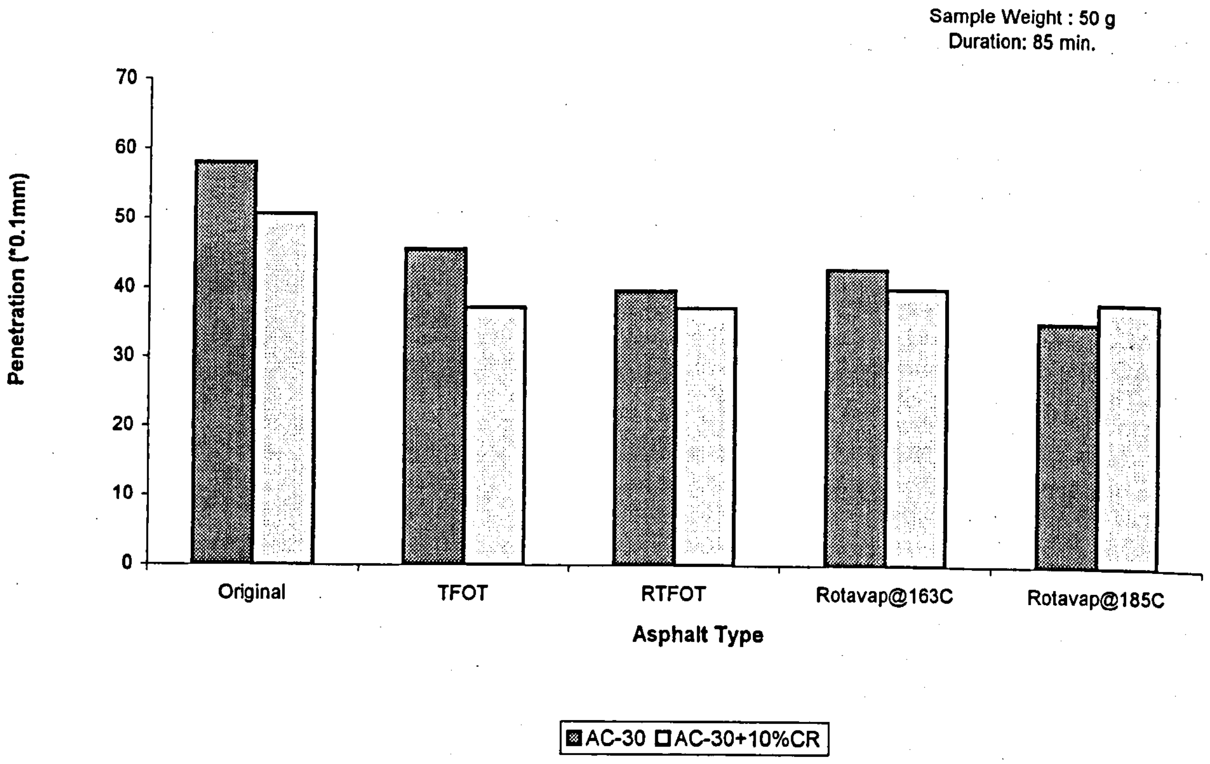

FIGURE 2 Comparison of penetration of asphalts after different aging processes.

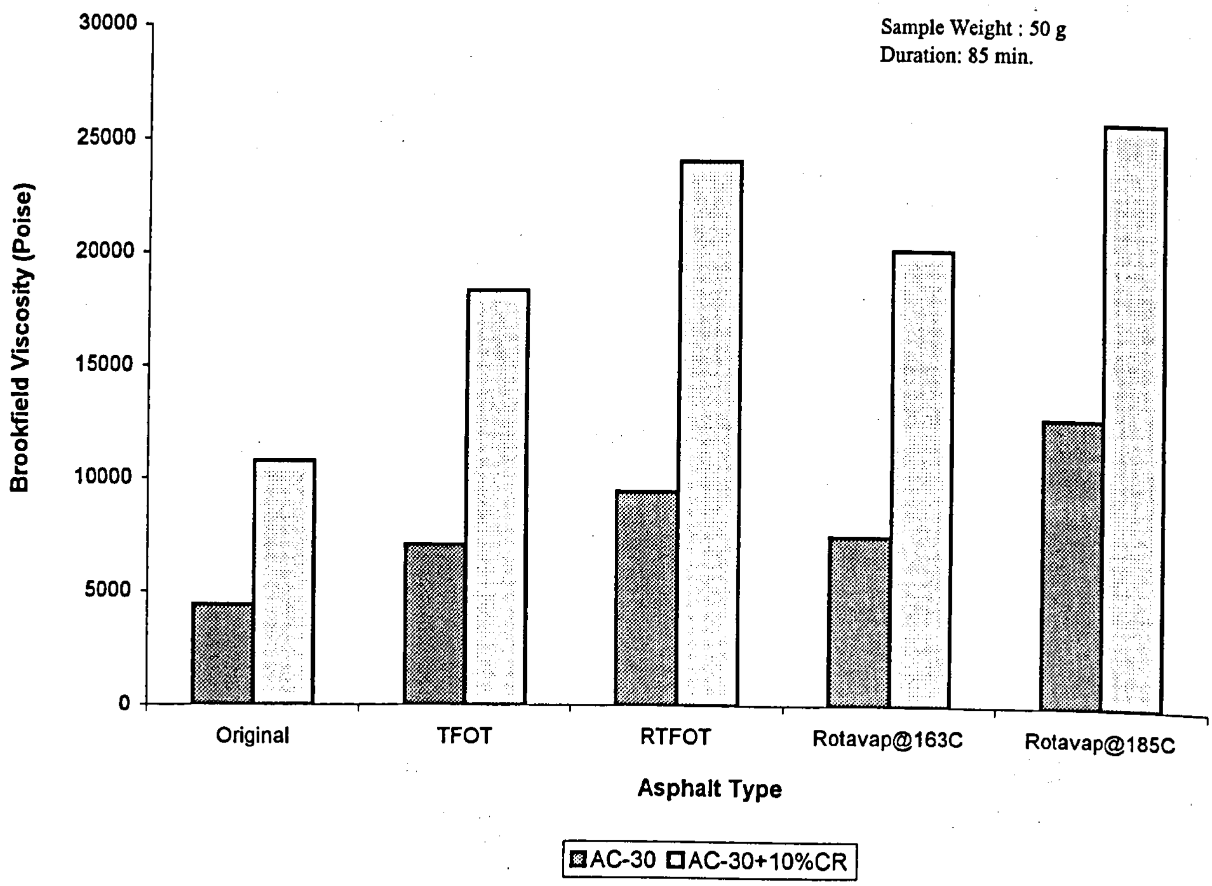

FIGURE 3 Comparison of Brookfield viscosity of asphalts after different aging processes.

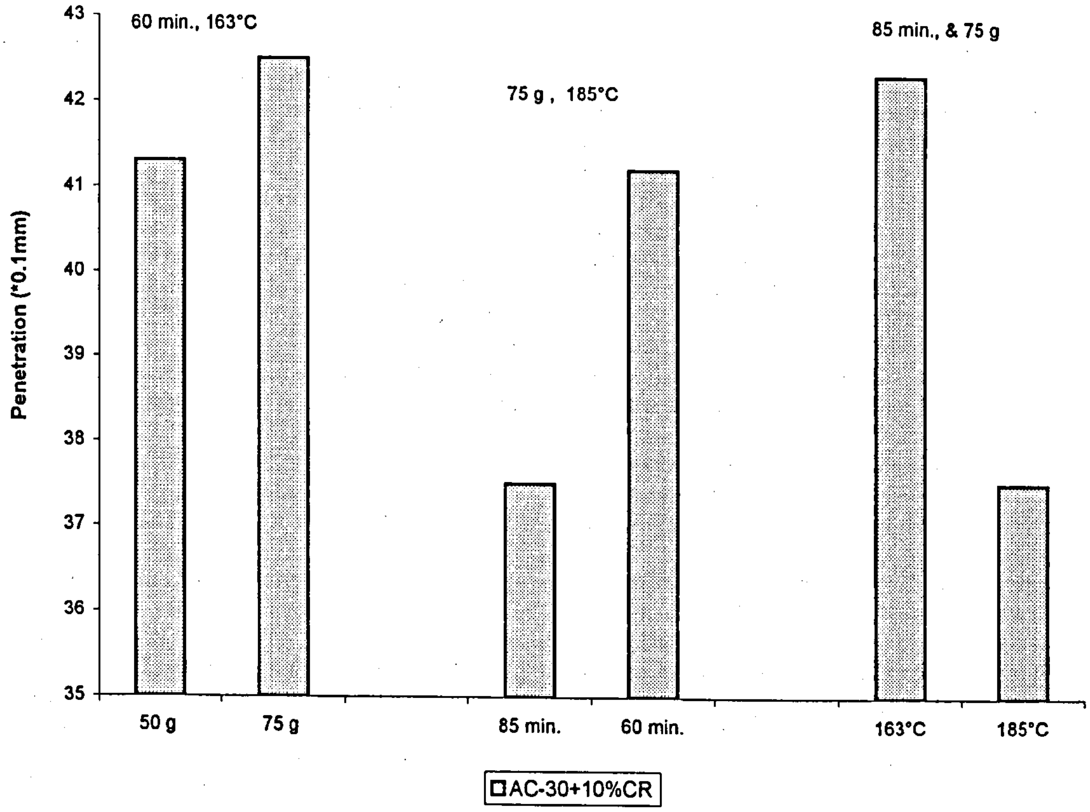

FIGURE 4 Effect of sample weight, aging duration, and process temperature on penetration of crumb rubber asphalts.

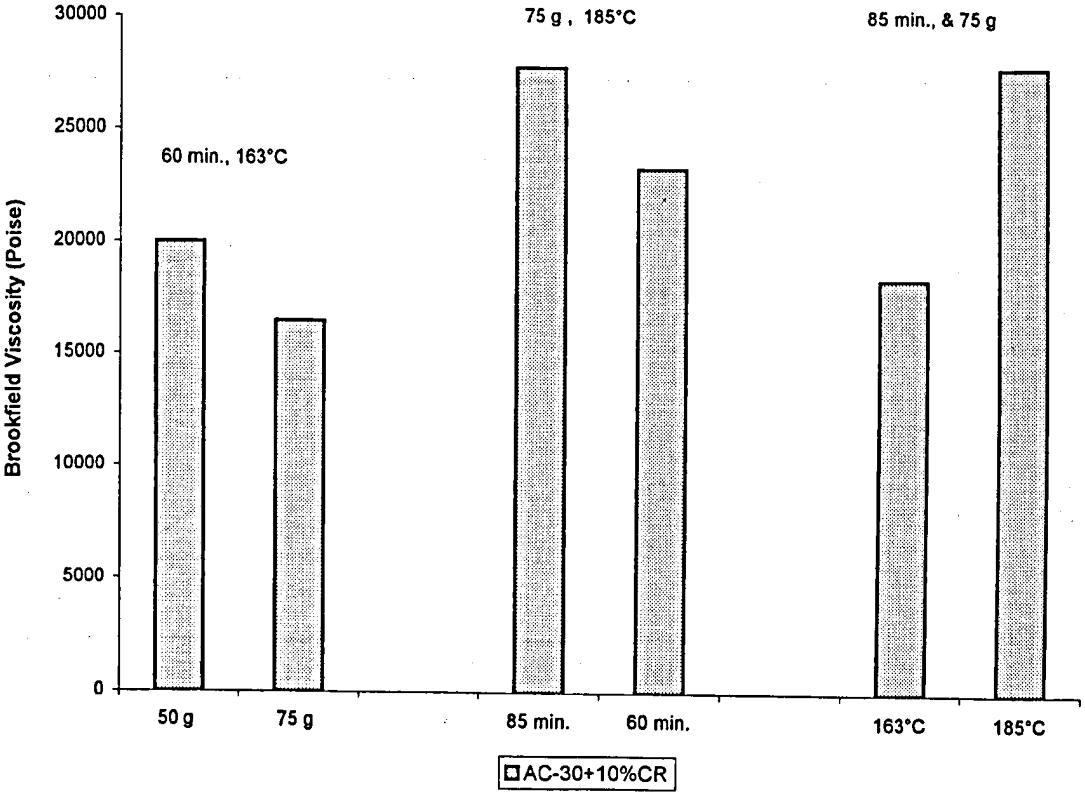

FIGURE 5 Effect of sample weight, aging duration, and process temperature on Brookfield viscosity of crumb rubber asphalts.

observation of the oil bath from the outside. The window material is 0.64-cm-thick tempered glass.

To determine improvement in temperature uniformity within the oil bath, temperature measurements were made with a resistance thermal detector (RTD) thermal probe at different locations within the oil bath, with and without the insulated box covering. Figure 6 shows plots of temperature versus vertical distance from the surface of the oil, along the centerline of the cylindrical oil container, with and without the box covering. It can be seen that, for the oil bath without the box covering, the temperature varied from 177°C at a depth of 8 cm to 142°C at a depth of 2 cm. For the oil bath with the box covering, the temperature stayed fairly constant at 181°C throughout the range of depth from 8 to 2 cm. For the case with the box covering, the temperature at the surface of the oil bath dropped only to 156°C, while for the one without the box covering, it dropped drastically to 95°C. It could be seen from these data that the use of the insulated box covering can substantially improve the temperature uniformity in the oil bath.

The temperature variation in the horizontal direction at a constant depth in the oil bath was also investigated with and without the box covering. Temperatures were measured with an RTD thermal probe at a constant depth of 8 cm, but at different horizontal positions within the oil bath. Figure 7 shows plots of temperature versus horizontal distance from the center at a constant depth of 8 cm in the oil bath. It can be seen that, at this fixed depth, the temperature stayed fairly constant, with or without the box covering. For the case without the box covering, the temperature stayed constant at 177°C. For the case with the covering, the temperature stayed at 181°C. These two different oil temperatures were obtained by using the same setting on the temperature control of the rotavapor apparatus. Thus, it is clear that the settings for temperature control to produce the same oil temperature would be different for the two test setups.

In the adopted test setup, the following settings of the rotavapor apparatus were used:

- The rotating flask is tilted at an angle of 45 degrees and submerged in the oil bath as far down as possible and such that the entire asphalt sample is below the oil level.
- The rotating speed of the flask is set at 60 rpm.
- The flow of air into the rotating flask is set at a rate of 4000 mL/min.
- The thermal probe that measures and controls the temperature of the oil bath is placed at a depth of 8 cm from the surface of the oil and near the wall of the container.

TESTING PROCEDURE ADOPTED

The adopted procedure for the modified rotavapor aging process consists of the following main steps:

1. The oil bath is turned on at least 1h before the start of the test to ensure that the specified process temperature has been reached.
2. The asphalt sample to be tested is heated to give sufficient fluidity before it can be poured into the flask.
3. The flask is placed on a scale, and a specified amount of asphalt is poured into the flask.
4. The flask is then left to cool for at least 1h to room temperature before it is placed into the rotavapor apparatus and submerged in the oil bath.

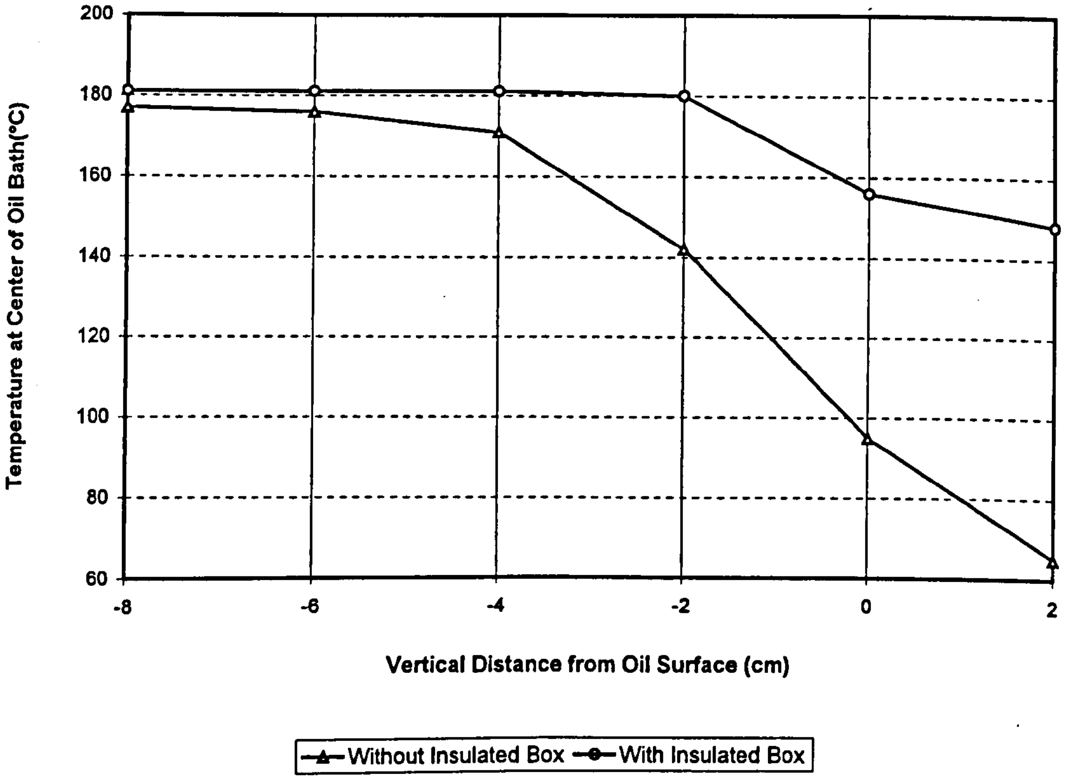

FIGURE 6 Temperature versus vertical distance from surface of oil bath of modified rotavapor aging apparatus with and without insulated box.

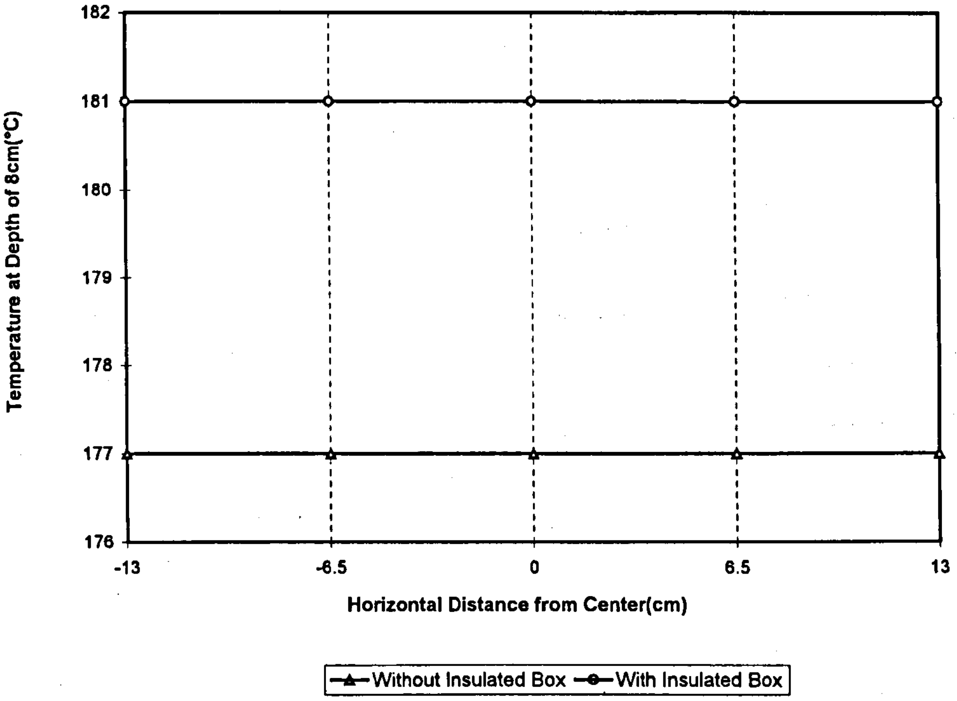

FIGURE 7 Temperature versus horizontal distance from center at depth of 8 cm in oil bath of modified rotavapor aging apparatus with and without insulated box.

5. The window of the insulating box is closed immediately. The rotation of the flask and the air flow into the flask are turned on immediately to start the test.

EVALUATION OF THE MODIFIED ROTAVAPOR AGING APPARATUS

A full factorial experiment was conducted to evaluate the performance characteristics of the modified rotavapor aging apparatus with the insulated covering. The following variables were incorporated in the factorial experiment:

- Two asphalt binders—AC-30 and AC-30 plus 10 percent CR with a nominal size No. 80 sieve.
- Three sample weights—50, 125, and 200 g.
- Three process durations—60, 85, and 110 min.
- One process temperature—163°C.

Standard TFOT and RTFOT aging processes were also applied to the same binders to compare with the modified rotavapor aging method. Two replicate samples were tested per combination of test parameters. After the asphalt binder had been aged in these processes, the following tests were run on each sample:

- Weight determination to determine weight change.
- Standard penetration test at 25°C (ASTM D5).
- Brookfield viscosity test at 60°C (ASTM D4402).

TEST RESULTS AND DISCUSSION

To observe the effects of process duration more easily, the average penetrations of the aged asphalt residues were plotted against process duration in Figure 8. It can be seen that the aging effect increases (as seen from the decrease in penetration) as process duration increases. Figure 9 shows the plots of the average penetrations of the aged residues versus sample weight. It can be seen that the aging effect decreases as sample weight increases.

The Brookfield viscosities of the residues were plotted against sample weight in Figure 10. It can be seen that the aging effect decreases (as seen from the decrease in viscosity) as the sample weight increases. Figure 11 shows the plots of the Brookfield viscosities of the residues versus process duration. The aging effect can be seen to increase as the process duration increases.

Figure 12 shows the comparison of the penetration of the aged residues after the various rotavapor processes with those of the TFOT and RTFOT residues. It can be seen that the modified rotavapor process at 163°C for 60 min using a sample weight of 50 g is closest to the TFOT and RTFOT processes in aging severity. It is slightly more severe than the TFOT, but slightly less severe than the RTFOT process. At this condition, the pure asphalt ages slightly more in the modified rotavapor process as compared with the TFOT residue. However, the CR-modified asphalt ages slightly less in the modified rotavapor process as compared with both the TFOT and RTFOT residues.

The original CR-modified asphalt was much harder (lower penetration) than the original pure asphalt. The aged CR-modified asphalt has a penetration very close to that of the aged pure asphalt. In the case of the insulated rotavapor process for a duration of 60 mins, the penetration of the aged CR-modified asphalt was even higher than that of the aged pure asphalt. This shows that the CR-modified asphalt generally ages less than the pure asphalt in terms of reduction in penetration.

The comparison of the Brookfield viscosity of the aged residues after the various rotavapor processes with those of the TFOT and RTFOT residues is shown in Figure 13. It can be seen that the

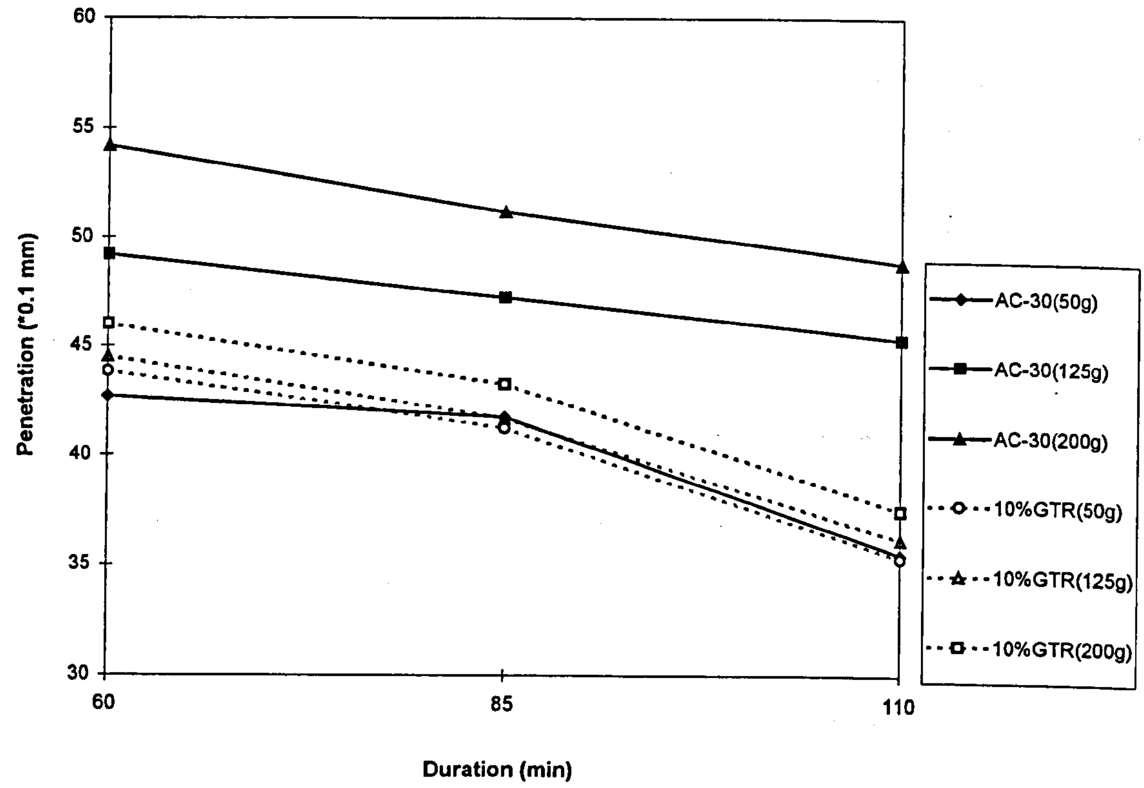

FIGURE 8 Penetration of residues after modified rotavapor process at 163°C versus process duration (GTR = ground tire rubber).

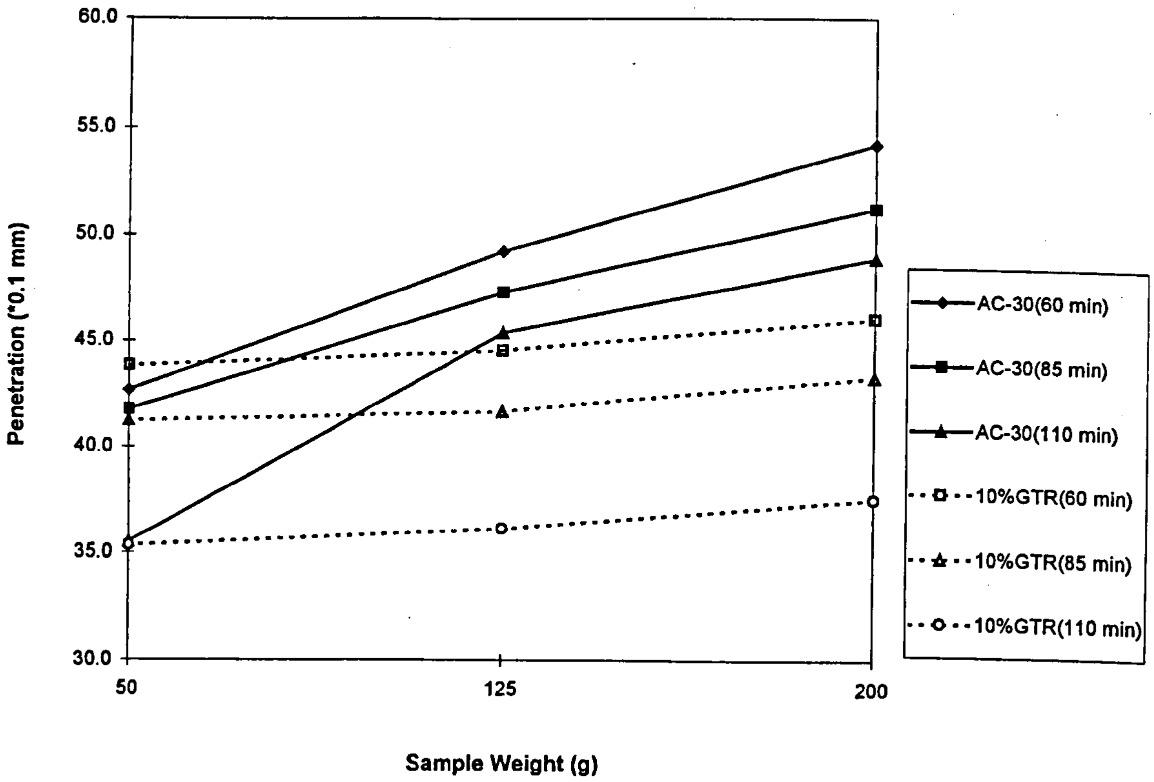

FIGURE 9 Penetration of residues after modified rotavapor process at 163°C versus sample weight.

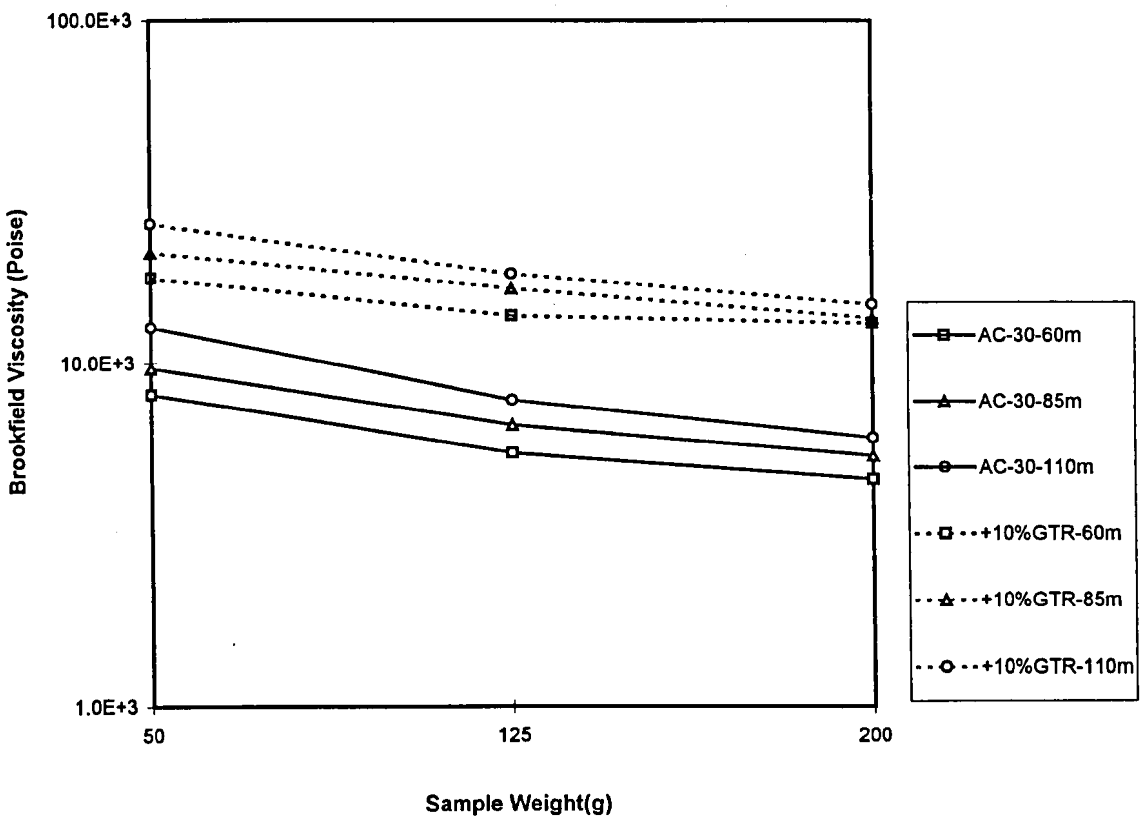

FIGURE 10 Brookfield viscosity of residues after modified rotavapor process at 163°C versus sample weight.

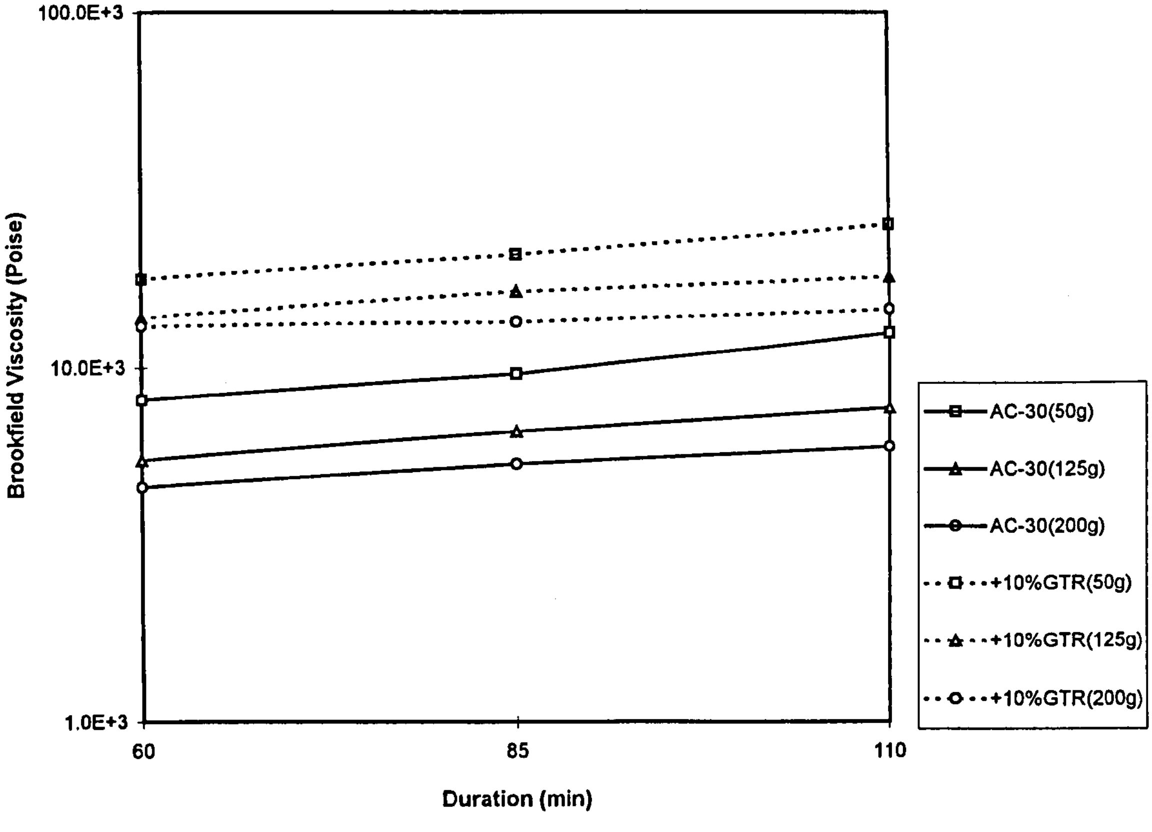

FIGURE 11 Brookfield viscosity of residues after modified rotavapor process at 163°C versus process duration.

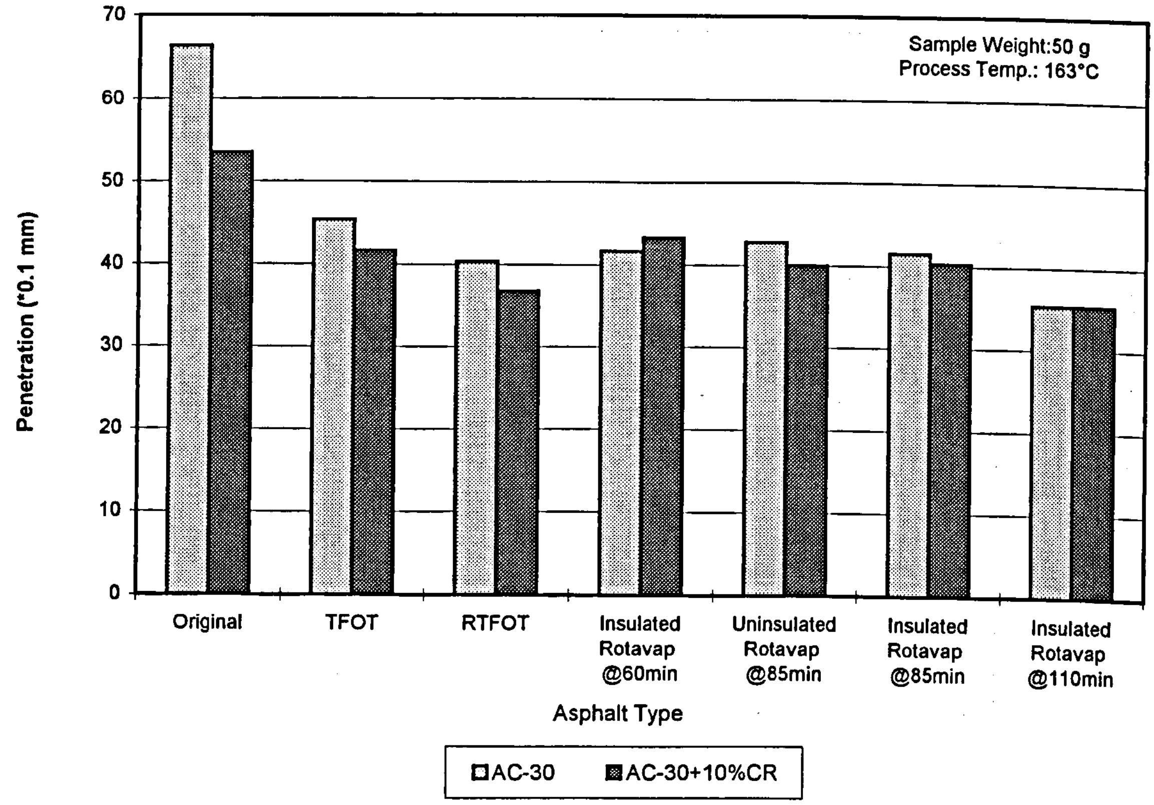

FIGURE 12 Comparison of penetration of aged residues.

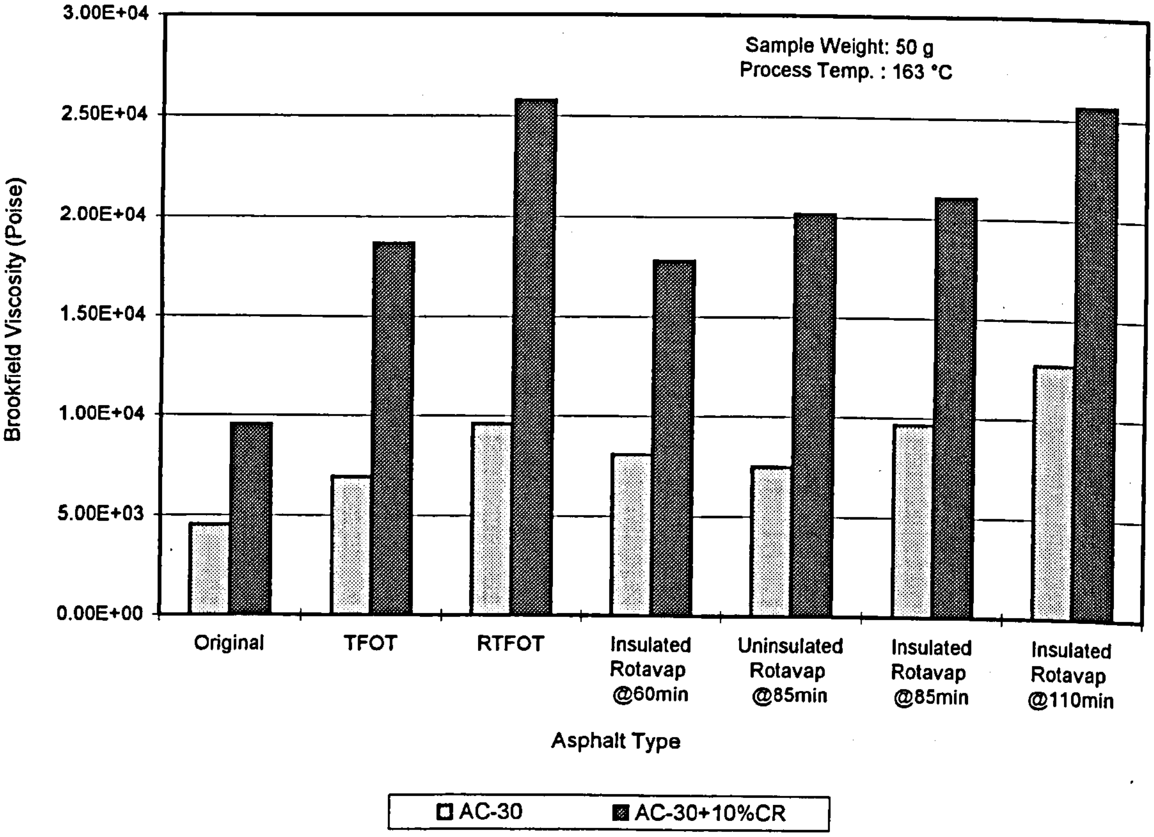

FIGURE 13 Comparison of Brookfield viscosity of aged residues.

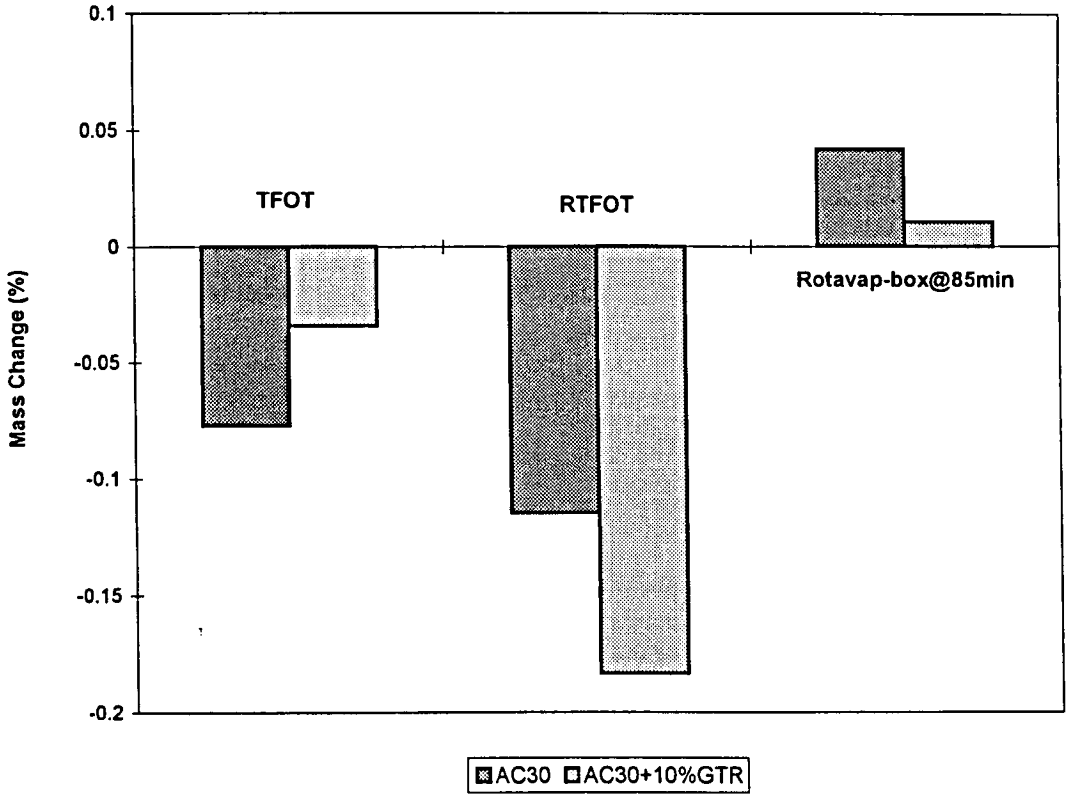

FIGURE 14 Comparison of mass change of asphalt after different aging processes.

comparison for the pure asphalts is different from that for the CR-modified asphalts. For the pure asphalts, the modified rotavapor process for 60 min using a sample weight of 50 g is closest to the TFOT, while the rotavapor process for 85 min using a sample weight of 50 g is closest to the RTFOT in aging severity. For the CR-modified asphalts, the modified rotavapor process for 60 min using a sample weight of 50 g is closest to the TFOT, while the rotavapor process for 110 min using a sample weight of 50 g is closest to the RTFOT in aging severity.

The average mass changes of the asphalt samples after the modified rotavapor process for 85 min using 50-g samples are compared with those of the TFOT and RTFOT residues in Figure 14. It can be seen that the TFOT and RTFOT residues experienced a mass loss, while the residue from the modified rotavapor process experienced a mass gain. These results agree in general with those reported by Zupanick and Baselice (*4*). The mass gain experienced in the rotavapor process could be explained by the oxidation process and small amount of volatile loss. The CR-modified asphalt is also seen to have less mass gain than the pure asphalt after the rotavapor process. More testing may be needed to validate this observation.

From Figure 14, it can also be seen that the RTFOT residues experienced more mass loss than the TFOT residues. This may be explained by the fact that there is more loss of volatiles in the RTFOT process due to the agitation of the asphalt samples in this test.

SUMMARY

The rotavapor apparatus used for recovery of asphalt was modified into an asphalt aging device as an alternative to the standard thin film oven and rolling thin film oven. To reduce the variation of aging condition due to variation of temperatures in the oil bath of the rotavapor apparatus, an insulated covering for the oil bath was constructed and used in this aging process. Evaluation results indicate that the aging severity of the modified rotavapor aging process is affected by variables such as process temperature, process duration, and sample weight. All these factors could be adjusted to produce the desired level of aging of asphalt samples. Because of the flexibility of controlling these variables in the modified rotavapor process, it appears that this method could be used to simulate the aging effects of the hot mix plant process effectively.

REFERENCES

1. Tia, M., B. E. Ruth, C. T. Chiu, S. C. Huang, and D. Richardson. *Improved Asphalt Cement Specifications to Ensure Better Performance.* Technical Report. Department of Civil Engineering, University of Florida, Gainesville, 1994.
2. Tia, M., B. E. Ruth, S. C. Huang, C. T. Chiu, and D. Richardson. *Development of Criteria for Durability of Modified Asphalt.* Technical Report. Department of Civil Engineering, University of Florida, Gainesville, 1994.
3. Zupanick, M. Comparison of the Thin Film Oven Test and the Rolling Thin Film Oven Test. *Journal of the Association of Asphalt Paving Technologists,* Vol. 63, 1994, pp. 346–372.
4. M. Zupanick and V. Baselice. Characterizing Asphalt Volatility. In *Transportation Research Record 1586,* TRB, National Research Council, Washington, D.C., 1997, pp. 1–9.

Publication of this paper sponsored by Committee on Characteristics of Bituminous Materials.