Effect of Crumb Rubber Modification on Short Term Aging Susceptibility of Asphalt Binder

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Abstract: Asphalt binder is subjected to aging during construction process and service life of asphalt pavement. Aging changes the chemical composition and physical properties of asphalt substantially which can lead to premature distresses on pavement. The changes in properties of the asphalt during the aging process is called aging susceptibility of asphalt. Aging of the asphalt is still a fact in the case of rubber modified binder. The effect of Crumb Rubber Modifier (CRM), interaction parameters, and exchange of the components on the aging behavior of the binder is not fully understood. In this study the effect of CRM modification on short term aging mechanism of asphalt is investigated through distinguishing between the effect of different activities, swelling and dissolution, of CRM on the aging process. Hence, the CRM binder and its extracted liquid phase were aged using Rolling Thin Film Oven. Physical properties of the neat and modified asphalt were measured and compared before and after the aging process and accordingly the aging susceptibility of each sample was calculated. Results show that CRM modification of asphalt significantly affects its aging susceptibility, however, the mechanism of its effect is different depending on the interaction conditions between asphalt and CRM.

DOI: 10.6135/ijprt.org.tw/2014.7(4).297 *Key words:* Asphalt rubber; Crumb rubber; Aging; Modified asphalt.

Introduction

One of the most critical stages in flexible pavement construction is the mixing and compaction process during which the asphalt binder goes through substantial aging, known as short term aging [1-2]. During this period, asphalt binder goes through different physical and chemical changes [1-2]. Comparing the properties of asphalt before and after aging reveals the aging susceptibility of asphalts. Asphalts with higher aging susceptibly are more prone to premature failures during the service life of the asphalt pavements.

The aging is still a fact in the case of modified asphalts. One of the common asphalt modifiers in industry is Crumb Rubber Modifier (CRM) due to its effect on enhancing the physical properties of asphalt as well as its environmental benefits through recycling scrap tires. The aging mechanism of asphalt as well as its interaction with CRM has been subject of numerous studies for several years.

Wu et al. investigated the changes in the micro-phases of asphalt binder during the aging. They showed that the multi-phase state of the neat asphalt binder tends to transform into a single-phase during the aging and the aging eliminates the borders between different phases [3]. Siddiqui et al. investigated the aging of asphalt binder using different analytical methods. They stated that carbonyl and sulfoxide groups increase considerably during the aging and there is a good correlation between their formation rate and the hardening rate of asphalt binders [4]. They also, stated that during the aging, aromatization and dehydrogenation of asphaltene occurs [5]. Isacsson et al. and Glover et al., in two different studies, investigated the effect of aging on chemical composition of asphalt, including the formation of functional groups like carbonyl and sufoxide, transformation of generic fractions of asphalt, and changes in the large molecular size components of asphalt [1, 6]. They stated that during the aging, the functional groups of asphalt oxidize and form carbonyl and sulfoxide groups. Also, part of the aromatic fraction of asphalt transform into the resins and part of the resins fraction transform into the asphaltene [1, 6]. In general, the aging of the asphalt is related to two main mechanisms; oxidization of functional groups in asphalt that increase its polarity, and volatilization of the light molecular components of the asphalt which function as peptizing agent. These two mechanisms lead to increase in the Asphaltene and resins concentration in asphalt and consequently increase the stiffness of the asphalt [1].

Aging of the CRM binder is much more sophisticated and complex than the neat asphalt due to its dynamic nature and effect of the time and temperature on enhancement of its physical properties [7]. CRM particles absorb aromatics in asphalt and swell up to 3 to 5 times their original size at elevated temperatures however, never reaching to the maximum level of swelling during the interaction period [8]. Since mixing and construction of asphalt is conducted at high temperatures, close to the asphalt and CRM interaction temperature, the swelling of CRM particles can continue during this time in addition to the other conventional aging mechanisms of asphalt binder and lead to more changes in final physical properties of CRM binder after its aging [9].

Moreover, depending on interaction parameters, CRM particles exchange their components, including antioxidants and polymeric components, with asphalt during the interaction. It is shown that each of these components individually affects the aging mechanism of asphalt. Antioxidants can retard the reactive groups of asphalt and prevent them from reacting with oxygen [10]. Also, polymeric chains act as retardant and inhibit the oxygen molecules from

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Note: Submitted December 31, 2013; Revised March 24, 2014; Accepted March 25, 2014.

penetrating into the asphalt and react with its functional groups. Moreover, it is shown that the polymeric chains may degrade during the aging and consequently neutralize part of the physical hardening happens due to oxidization and volatilization of asphalt components [10-11].

In this study, the effect of CRM particles and their exchange of components with asphalt were investigated on the aging susceptibility and physical aging mechanism of the Modified Asphalt (MA). In this regards, the MA and its Liquid Phase (LP); interacted CRM is removed, called MA-LP throughout this research, were prepared at different interaction conditions and aged through Rolling Thin Film Oven (RTFO). The aging susceptibility of different samples was calculated comparing their rutting parameter ($\frac{G^*}{\sin \delta}$) before and after aging [1]. Then, the effect of CRM on volatilization of light molecular components of the samples during the aging was studied by conducting Thermo-Gravimetric Analysis on MA and MA-LP samples.

The long term aging of the MA is not considered in this research as the mechanism of the long term aging of the asphalt is different from the one happens at short term aging stage and it is out of scope of this work.

Materials and Methods

Materials

Two asphalts from two different sources with the same PG grade of 58-28 were utilized in this research to consider the effect of asphalt source in the results. Asphalt batches denoted NF and M were provided by Flint Hill Resources and Murphy Oil Corporation, respectively. Also, to consider the effect of CRM composition, two different CRM batches were utilized in this research. Both CRM batches were produced by ambient method and were provided by Liberty Tire Recycling. One CRM batch was produced from Truck Tire resources (TR) and the other was produced from passenger car tires (WTG). The CRM particle size, in all interactions, was controlled to be smaller than the mesh#40 (0.422 mm) and larger than the mesh#60 (0.251 mm), according to US standard system, unless otherwise stated. In case of different CRM size, the CRM size is presented in the code of interaction, showing the upper and lower sieve size. For instance "(20-30)" in "NF-TR-(20-30)-15%-10Hz160C" indicates that the CRM particles in this interaction passed Sieve#20 (0.853 mm) and retained on sieve#30 (0.599 mm). The properties of the materials are listed in Table 1.

Table 1. Properties of the Asphalt and CRM.

Physical Properties @ 58°C Asphalt Type PG Grade $\frac{G^*}{\sin\delta}$ (Pa) G* (Pa) G'(Pa) G" (Pa) δ (Degree) Aging Susceptibility (Unit-less) Flint Hill (NF) PG58-28 1232 53.47 1230 87.51 1643.2 2.6 PG58-28 1640 103.3 1637 86.39 1233.2 Morphy (M) 2.6 Process Method % Acetone Extract %Carbon Black CRM Type Source %Polymer %Other TR Truck Tire Ambient 3.85 57.42 30.84 7.89 WTG Passenger Car Tire Ambient 5.59 55.45 31.41 7.55

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Interactions

All interactions were conducted for 2hrs, except otherwise stated, using 1200±100 g of asphalt in 1 gal paint cans. An aluminum heating mantle (100B TM634) attached to a bench top temperature controller and a J-type thermocouple was utilized to control the interaction temperature. Nitrogen gas was applied on top of all interactions to prevent any oxidization. Table 2 shows the list of interactions, their related code and parameters. The interaction parameters were selected based on the results of the previous studies by this research group to control the dissolution of CRM in asphalt and develop a set of samples with a wide variety of CRM dissolution percentages [8, 12]. The reliability of all interactions and testing was verified by running replications on limited number of samples based on feasibility.

CRM Extraction Method

To measure the dissolution of the CRM in asphalt before and after aging, the CRM extraction method, as explained in previous studies by the authors, was performed in this research [12]. In this method, 10 g of modified asphalt were diluted in Toluene, drained through a mesh#200 (75 μ m) and washed with extra Toluene until the filtrate became colorless. The retained particles were dried in the oven at 60°C for 12 hrs to assure removal of all solvent residues.

Extraction of Liquid Phase of Modified Asphalt

To investigate the effect of exchanged components between CRM and asphalt on aging mechanism of the MA, the liquid phase of the MA samples was extracted before and after aging of the MA. To obtain the liquid phase, the required amount of MA was heated to 165°C and drained through mesh#200 (75 μ m) in the oven at 165°C for 25 min. Immediately, the extracted liquid phase (MA-LP) was stored at -12°C to prevent any unwanted aging or reaction.

Dynamic Shear Rheometer (DSR)

Dynamic Shear Rheometer from Bohlin Instruments CVO (Worcestershire, UK) was used for viscoelastic analysis. All tests were performed using 25 mm diameter parallel plates at 58°C and 1.59 Hz to follow the Superpave guidelines. The gap between plates for MA samples was selected to be 2 mm, the minimum gap size that doesn't affect the results due to the presence of CRM particle [8, 12]. For neat asphalt and the liquid phase, where there was no CRM particles, the gap size was selected to be 1 mm. To avoid nonlinear

Interaction Name	AC Type	CRM Type	CRM Size	CRM Concentration (%)	Interaction Temp (°C)	Mixing Speed (Hz)	CRM Dissolution (%)	Aging Susceptibility (Unit-less)
NF-TR-5%-10Hz160C	NF	TR	(40-60)	5	160	10	10.8	2.4
NF-TR-10%-10Hz160C	NF	TR	(40-60)	10	160	10	12.2	2.2
NF-TR-(20-30)-15%-10Hz160C	NF	TR	(20-30)	15	160	10	11.1	2
NF-TR-15%-10Hz160C	NF	TR	(40-60)	15	160	10	11	1.7
NF-TR-(80-200)-15%-10Hz160C	NF	TR	(80-200)	15	160	10	13	1.7
NF-TR-15%-10Hz190C	NF	TR	(40-60)	15	190	10	13	1.7
NF-TR-15%-30Hz190C	NF	TR	(40-60)	15	190	30	25.9	1.8
NF-TR-15%-50Hz190C-60min	NF	TR	(40-60)	15	190	50	35	1.7
NF-TR-15%-50Hz190C	NF	TR	(40-60)	15	190	50	58.2	2
NF-TR-15%-50Hz220C	NF	TR	(40-60)	15	220	50	86.5	2.3
NF-WTG-15%-10Hz160C	NF	WTG	(40-60)	15	160	10	NA	2
NF-WTG-15%-50Hz220C	NF	WTG	(40-60)	15	220	50	NA	2.3
M-TR-15%-10Hz160C	Μ	TR	(40-60)	15	160	10	NA	2.1
M-TR-15%-50Hz220C	М	TR	(40-60)	15	220	50	NA	2.6

Table 2. List of Interactions and Corresponding Parameters.

NA = Not Available

viscoelasticity, all tests were performed in a strain control mode with strain less than 1%.

Short Term Aging

In this study all samples were aged through RTFO, ASTM D2872-12 standard, except otherwise stated. To assure a complete flow of the samples in the RTFO bottles (specially in the case of MA samples), the suggested method by Bahia et al. was applied [7]. Based on this method, a stainless steel rod by the size of 127 mm by 6.4 mm was placed in each bottle to ease the flow of the samples and prevent them from rolling out, which were observed during the preliminary studies by the authors and also reported in other literatures [7, 13]. For consistency the rods were used for all samples, including the unmodified asphalt and modified asphalts. For reliability, at least two replication of RTFO was conducted on each sample.

In this study, the aged MA samples have been noted as MA-RTFO and the aged MA-LP (the liquid phase of MA that was extracted before aging) have been noted as MA-LP-RTFO. Moreover, after aging the MA, its liquid phase was extracted from the whole matrix and denoted MA-RTFO-LP throughout this research. Note that in the coding that, the sequence of MA, LP, and RTFO defines the sequence of the job. For instance, MA-RTFO-LP indicates that the modified asphalt was aged first and then its liquid phase was extracted. But, MA-LP-RTFO indicates that the liquid phase of modified asphalt was extracted first (before aging) and then the liquid phase was aged through RTFO.

To investigate the age hardening of the samples the following equation was utilized:

$$Aging Susceptibility = \frac{\frac{G^*}{\sin \delta_{after aging}}}{\frac{G^*}{\sin \delta_{before aging}}}$$
(1)

In Eq. (1), G* represents the complex modulus of asphalt and δ represents the phase angle of the asphalt. The $\frac{G^*}{\sin \delta}$ in Eq. (1)

represents the rutting parameter of the samples before and after aging. For the aging susceptibility of the MA, the $\frac{G^*}{\sin \delta}$ of the MA after RTFO aging was divided by its $\frac{G^*}{\sin \delta}$ before aging. For the aging susceptibility of the MA-LP, the $\frac{G^*}{\sin \delta}$ of MA-LP-RTFO was divided by the $\frac{G^*}{\sin \delta}$ of MA-LP and finally for the aging susceptibility of MA-RTFO-LP, its $\frac{G^*}{\sin \delta}$ was divided by the $\frac{G^*}{\sin \delta}$ of MA-LP.

Thermo-Gravimetric Analysis (TGA)

Thermo-Gravimetric analysis is simple yet common experiment which measures the weight loss of the samples as a function of temperature. This experiment extensively and its applications in asphalt rubber industry is explained in other literature of the authors [14-15]. During aging of the asphalt, part of its aromatics volatilize and result in higher concentration of resins and asphaltene and consequently higher stiffness of aged asphalt. In order to accurately investigate the effect of CRM modification on aromatics volatilization, TGA analysis was conducted on modified asphalt (MA) samples and their liquid phase (MA-LP). TGA is a simple but useful method to measure the mass loss of the samples as a function of temperature. All TGA tests were performed by using TA Instruments' Q500 TGA in isothermal mode. In this method, 45±5 mg of sample was loaded on an aluminum sample holder and heated to 163°C at a heating rate of 20°C/min and then the temperature was kept constant for 85 min. The testing temperature and duration was selected in order to simulate the RTFO aging conditions. The total mass of the sample was monitored by the instrument during this period and any mass loss was recorded.

Results and Discussions



Fig. 1. Effect of Short Term Aging on CRM Dissolution.

Effect of Short Term Aging on CRM Dissolution

Studies by this research group show that CRM particles partially dissolve into the asphalt matrix and release part of their components into the asphalt matrix, mainly depending on interaction temperature [12]. At very low interaction temperatures (i.e. 160°C), the dissolution of CRM is minimized and limited to its oily components. However, at higher interaction temperatures (above 190°C) the dissolution extends to polymeric components of CRM. In this section, the effect of RTFO aging on CRM dissolution is studied by comparing the dissolution percentage of CRM before and after aging, Fig. 1.

Results in Fig. 1 show that the CRM dissolution continues during the aging, however, it diminishes by increasing the original CRM dissolution (before aging). In other words, the more intact CRM particles before aging, the more prone they are to disintegrate during the short term aging. Also, the results show that CRM particles with smaller size are subject to more disintegration during the aging than the larger CRM particles.

Effect of CRM Size and Concentration

To investigate the effect of swollen CRM particles on short term aging of MA, several MA samples with different CRM concentrations and CRM sizes were produced. To limit the activity of the CRM to swelling and minimize the level of its dissolution, the interaction condition was controlled at 160°C and 10Hz and the interaction time was limited to 2hrs. Fig. 2 shows the changes in physical properties of MA and its liquid phase before and after aging and their aging susceptibility as a function of CRM concentration (Fig. 2a and 2b) and CRM size (Fig. 2c and 2d).

In Fig. 2a and 2c, the aging susceptibility is calculated based on Eq. (1). The results in these figures show that the aging susceptibility of the MA-LP doesn't change as a function of the CRM concentration or CRM size, even though increasing the CRM concentration increases the absorption of light molecular components of asphalt and consequently enhances the physical properties of the MA-LP before aging, shown in Fig. 2b. This



Fig. 2. (a) Aging Susceptibility (b) Rutting Parameter of Samples as a Function of CRM Concentration (c) Aging Susceptibility (d) Rutting Parameter of Samples as a Function of CRM Size.

indicates that absorption of light molecular weight components of asphalt by CRM particles has no remarkable effect on the extent of its age hardening and consequently its aging susceptibility.

Fig. 2a shows that aging susceptibility and rutting parameters of the MA-RTFO-LP increases much faster than the ones for MA-LP-RTFO as a function of CRM concentration. This indicates that the liquid phase of the MA ages at a higher rate and to a higher extent in the presence of the CRM particles. Therefore, it can be concluded that in addition to the conventional aging mechanisms of neat asphalt (oxidization, volatilization, and polymerization) other aging mechanisms are also involved in the case of MA which is discussed in subsequent sections.

In Fig. 2, the aging susceptibility and rutting parameter of the MA-RTFO-LP increases by increasing the CRM concentration (Fig. 2a and 2b), and decreases by decreasing the CRM size (Fig. 2c and 2d). This indicates that two phenomena are involved in age hardening process. The first phenomenon is the continued absorption of light molecular weight components of asphalt by CRM particles which intensifies by increasing the CRM concentration, Fig. 2a and 2b. The second phenomenon is the oxidization and volatilization of the liquid phase during the aging which is expected to get reduced by improving the CRM dispersion in asphalt through reducing its particle size, Fig. 2c and 2d. These are further investigated in the subsequent sections.

Moreover, in Fig. 2a, it can be seen that even though the aging susceptibility of the MA-RTFO-LP increases by increasing the CRM concentration, the aging susceptibility of the MA itself decreases. This is attributed to the dominant role of CRM particles in defining the physical properties of the MA at high CRM concentrations which is due to their marginal susceptibility to aging.

Effect of CRM Dissolution

In this section, the effect of CRM dissolution on short term aging of the modified asphalt is investigated. In this regard, the dissolution of CRM in asphalt was controlled through monitoring the interaction conditions. Fig. 3 shows the changes in aging susceptibility and physical properties of samples as a function of CRM dissolution percentage.

As Fig. 3a presents, the rutting parameter of the MA deteriorates by increasing the CRM dissolution, however, the complex modulus of the MA-LP increases slightly first and then deteriorates, which are both in accordance with the previous studies by this research group [8, 12].

Fig. 3b shows that the aging susceptibility of the MA-LP and MA-RTFO-LP decreases significantly by increasing the CRM dissolution and finally reaches to the aging susceptibility of the MA. The decrease in aging susceptibility of the MA-LP can be attributed to the partial release of the CRM components in asphalt. These components are proven to interfere with age hardening mechanisms that are happening to the asphalt [16]. The decrease in aging susceptibility of the MA-RTFO-LP as function of CRM dissolution can be explained by the reduction in swelling capacity of the remaining CRM particles after partial dissolution and also the effect of released components of CRM into the asphalt matrix as explained in the case of the MA-LP.

Moreover, Fig. 3b shows that the aging susceptibility of the MA



Fig. 3. (a) Rutting Parameter (b) Aging Susceptibility as a Function of CRM Dissolution.

stays relatively constant by increasing the CRM dissolution up to 35% and then starts to increase slightly by further CRM dissolution and reaches to the aging susceptibility of the MA-LP and MA-RTFO-LP. This indicates that the dominated role of CRM particles in defining the mechanical behavior of MA matrix diminishes by CRM dissolution, and the physical properties of the MA and its aging mechanism are being defined by the liquid phase of the MA rather than whole matrix (asphalt plus CRM particles).

Effect of Aging Temperature

CRM modification of asphalt remarkably increases its high temperature viscosity which drastically affects the handling of the binder as well as the mixing and compaction process of hot mix asphalt. Therefore, in different research works and technical reports it has been suggested to increase the temperature to remediate this problem [17]. This increase in temperature highly affects the age hardening of the modified binder. To investigate the effect of the increased mixing temperature on aging of the MA, the standard RTFO test was conducted at four different temperatures, 163°C, 170°C, 180°C, and 190°C and the results are presented in Fig. 4.

Fig. 4a shows that increasing the aging temperature significantly affects CRM particles' integrity in the asphalt and accelerates their dissolution during aging. Fig. 4b shows that the rutting parameter of the MA-LP-RTFO as a function of aging temperature increases significantly, whereas, the rutting parameter of the MA and

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Fig. 4. (a) Dissolution of CRM During the Aging Process (b) Rutting Parameter (c) Aging Susceptibility as Function of Aging Temperature.

MA-RTFO-LP increases much slower and then starts to decrease by increasing the aging temperature to 190°C. Fig. 4c shows the same trend for aging index of the three components.

Increasing the aging temperature leads to disintegration of CRM particles in asphalt during aging and consequently desorption of the light molecular components of asphalt back into its matrix. These two phenomena, in spite of the hardening that happens due to faster oxidization of the asphalt binder, presented in next section, result in deterioration of the rutting parameter of the MA and MA-RTFO-LP, as presented in Fig. 4b. The reduction in rutting parameter leads to lower aging susceptibility, in Fig. 4c.



Fig. 5. Effect of CRM on Mass Loss of Asphalt During the Aging.

Moreover, Fig. 4c shows that the aging susceptibility of MA-LP-RTFO, that aged at 190°C, is much lower than the original asphalt, aged at same condition. This indicates that regardless of presence of the CRM particles in the matrix, released components of the CRM in the asphalt, even at very low original CRM dissolutions, improve its aging susceptibility and prevent it from oxidization.

All these results illustrate that increasing the temperature is not an appropriate remediation to resolve the high viscosity issue of MA for handling and construction purposes.

Effect of CRM on Oxidization and Mass Loss

The effect of CRM modification on mass loss of the samples during the aging was investigated by conducting TGA tests and the results are presented in Fig. 5.

The results in Fig. 5 show that all MA samples and their liquid phase show slightly lower mass loss at aging conditions comparing to the original asphalt, and as the interaction conditions (temperature and mixing speed) increases, the mass loss of the samples relatively decreases. This can be attributed to the partial volatilization of the light molecular components of asphalt during the interaction and also their absorption by CRM particles.

Moreover, comparing the results of the interaction "NF-TR-15%-10Hz160C" and its liquid phase "NF-TR-15%-10Hz160C (MA-LP)" in Fig. 5 indicates that the presence of the CRM particles has no significant effect in preventing the aromatics of asphalt from volatilization.

Effect of Asphalt and CRM Type

In order to investigate the effect of the asphalt and CRM source on the general trends that were observed in this research, a limited number of interactions were conducted using different sources of the CRM and asphalt as stated in Table 1. The effect of changing the sources of the material on the aging mechanism of the MA is presented in Fig. 6.

Results in Fig. 6 indicate that different types of CRM and asphalt lead to different values in aging susceptibility and rutting parameters but the general trends that were explained in this study stays the same for all material sources. The differences in the values



Fig. 6. Effect of Asphalt Type and CRM Type on Aging Index of (a) MA (b) MA-LP-RTFO (c) MA-RTFO-LP.

can be attributed to different swelling capacity of CRM samples and also to different stiffness of the original asphalt samples under study.

Conclusions

The effect CRM dispersion in asphalt matrix and the exchange of components between asphalt and CRM were studied on the short term aging mechanism of modified asphalt (MA). In this respect the MA samples and their liquid phase (MA-LP) were aged through RTFO. The physical aging susceptibility index was calculated using DSR. Also, to study the effect of CRM on mass loss of the asphalt during the aging, TGA test was conducted.

The results show that CRM particles have no significant role in depressing the volatilization of aromatics of asphalt during the aging as the MA samples and their liquid phase show no remarkable difference in mass loss.

The results also reveal that the CRM particles, when not dissolved, continue to absorb the aromatics and light molecular weight components of asphalt which leads to significant stiffening of the liquid phase of modified asphalt that is extracted after the aging (MA-RTFO-LP). But the dominant role of CRM particles in defining the mechanical behavior of the MA, especially at high CRM concentrations (i.e. 10%, 15%), keeps the aging susceptibility of the MA matrix lower than the unmodified asphalt.

Partial dissolution of the CRM particles into the asphalt matrix leads to superior stability of the whole matrix and its liquid phase as the CRM particles lose their absorption capacity and are unable to swelling during the aging process. This will help to assure the consistency of the nature of the binder all through the mixing and compaction process of the hot mix asphalt.

Finally, the results show that increasing the mixing and compaction temperature is not an appropriate solution to resolve the high viscosity issue of the MA. Results show that increasing the temperature leads to considerable disintegration of CRM particles which significantly changes the nature of the modified asphalt.

Acknowledgement

This material is based on the work supported by the National Science Foundation under Grant No. 0846861. Any opinions, findings, and conclusions or recommendations expressed in this material are those of the writer(s) and do not necessarily reflect the views of the National Science Foundation.

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