AGING OF BITUMINOUS BINDERS – LABORATORY TESTS AND FIELD DATA

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ABSTRACT

A comprehensive literature study on road durability has indicated that the aging susceptibility of bituminous binders in surface layer is a key parameter determining the life time of an asphalt pavement. To simulate bitumen aging in laboratory, several test methods are currently used, including RTFOT, PAV, and RCAT (Rotating Cylinder Aging Test). In this paper, the aging of bituminous binders (unmodified and SBS polymer modified) were studied using (i) RTFOT, followed by PAV at 100°C for 8 to 48h; (ii) RTFOT, followed by PAV at 75°C for 48 to 220h; and (iii) RCAT at 163°C for 4h, then continued at 90°C for 17 to 140h. For field core sampling, several roads of different ages were selected from the Swedish LTPP (Long Term Pavement Performance) database. The study showed that RCAT at 163°C/4h, proposed as a short-term aging test, was similar to RTFOT. The long-term aging tests PAV (100°C, 20h), PAV (75°C, 120h) and RCAT (90°C, 140h) were also more or less equivalent when applied to unmodified bitumens. However, for polymer modified bitumens, these tests were significantly different with respect to the resulted aging kinetics. As regards aging in the field, it was found that after 10 – 30 years service on the road, the extracted bitumens displayed a relatively low degree of age-hardening, to which equivalent laboratory aging durations were much shorter than that being standardized or proposed. The low degree of field aging may be attributed to low air voids in the asphalt mixtures and/or prevention of bitumen oxidation by surface sealing or overlaying. Field aging also considerably differed from laboratory aging in terms of the formation of sulfoxides and carbonyl functionalities in bitumen. Compared to the laboratory aging, much higher level of sulfoxides but lower level of carbonyls was found for the binders aged in the field. This suggests that oxidation mechanisms in the field may not be the same as in the laboratory aging tests.

Keywords: aging, laboratory testing, field durability, polymer modified bitumen

1. INTRODUCTION

The aging of bituminous binders is one of the key factors determining the lifetime of an asphalt pavement. The process of aging involves chemical and/or physical property changes that usually make bituminous materials harder and more brittle, thus increasing risk of pavement failure. The aging-related pavement failure modes include cracking (thermal or traffic induced) and ravelling. Cracks on pavement surface may increase aging of the binder because of increased exposure area to atmospheric oxygen.

In general, bitumen aging takes place in two stages, namely short-term aging at high temperature during asphalt mixing, storage and laying, and long-term aging at ambient temperature during in-service. The mechanisms of aging include oxidation, evaporation and physical hardening. Physical hardening is a reversible process, which changes the rheological properties of bitumen without altering its chemical composition. At ambient temperatures, physical hardening normally is very slow, but it can speed up at low temperatures. For bituminous binders, loss of volatile components (evaporation) is also considered as an aging mechanism. However, today’s penetration grade bitumens are relatively involatile, thus during pavement in-service this type of aging is negligible.

As a principal mechanism, oxidative aging is an irreversible chemical reaction between components of bitumen and oxygen. It may occur through different reactions, such as photo-oxidation and thermal oxidation [1]. The light (mainly ultraviolet, UV) catalyzed reaction occurs rapidly and generally takes place within the top 5 μm of the exposed binder film, since bitumen is a good light absorber [1] [2]. In spite of the limited penetration into bitumen, the photo-oxidation induced aging may have an influence on durability, particularly for some polymer modified binders [3]. Nowadays, most of aging on the road is still regarded as thermally induced. The rate of thermal oxidation of bitumen is approximately doubled for every 10°C rise in temperature. Thus aging rate in service depends to a large extent on pavement temperature [4] [5]. Certainly, the oxidative aging is influenced by the chemical nature of bitumen. It has been shown that different bitumens have very different increase of viscosity with aging time, and the temperature-dependence of aging kinetics is strongly dependent on the bitumen [6] [7] [8].

Another important factor affecting bitumen aging on the road is the void content of asphalt mixture. Much work has indicated that asphalt mixtures of low voids show a low degree of bitumen aging while higher void content facilitates the aging process [1] [4] [9] [10] [11]. Presumptively, void content determines the rate of aging by controlling oxygen access to the bitumen. Thus, the oxidative aging of bitumen as function of depth in the pavement is closely related to void content of the mixture.
To simulate field aging in laboratory, different types of test may be used, including conduction of accelerated aging on bituminous binders, loose asphalt mixture, or on compacted asphalt specimen. For bituminous binders, there are three European standardized tests [12] for short-term aging at high temperatures, namely Rolling Thin-Film Oven Test (RTFOT, EN 12607-1), Thin Film Oven Test (TFOT, EN 12607-2), and Rotating Flask Test (RFT, EN 12607-3). These tests reasonably simulate aging particularly during mixing process in an asphalt mixing plant.

For long-term aging during in-service, laboratory simulation is rather difficult. Ideally, a laboratory test should be able to predict chemical and physical property changes in the bitumen which occur after certain years on asphalt pavement. This may be achieved by conducting an aging test at artificially severe conditions, e.g. at temperatures higher than pavement service temperature and at pressures higher than ambient pressure. Two European standardized long term aging tests are Pressure Aging Vessel (PAV) [13] and Rotating Cylinder Aging Test (RCAT) [14]. Although numerous investigations have been carried out, solid data, especially field data for different types of binders under different climatic conditions, are still not sufficient to support if these laboratory aging tests are relevant or if natural aging occurred in the pavement can be properly predicted. It is also believed that aging of bitumen at a higher temperature may be fundamentally different from aging at lower temperature that is more accurately simulating pavement temperature [5].

The primary objectives of this study are to compare how different aging tests (RTFOT, PAV and RCAT) under various conditions relate to field aging. Based on the Swedish LTPP (Long Term Pavement Performance) database [15], several roads of different ages were selected to drill asphalt cores and to extract bitumens from the asphalt cores. Accordingly, fresh unmodified and SBS polymer modified bitumens were prepared for laboratory aging. Characterization of binders was performed using conventional tests (penetration, softening point), rheological measurements with dynamic shear rheometer (DSR), as well as by Fourier transform infrared spectroscopy (FTIR).

2. EXPERIMENTAL

2.1 Laboratory aging

Three types of binders were selected and prepared for this study. The selection was based on the field samples collected (Cf. Section 2.2) and was an attempt to make fresh samples of the same type of bitumen as used in the construction of the roads. The binders included a viscosity-grade bitumen A120 (penetration 220 dmm, softening point 35.3°C), a penetration-grade bitumen B85 (penetration 76 dmm, softening point 45.5°C), and a polymer modified bitumen PMB20 with 6% linear SBS (penetration 98 dmm, softening point 95°C). According to today’s European specifications, A120 and B85 may be classified as 160/220 and 70/100, respectively.

The binders were aged by different procedures under different conditions as follows:

- RTFOT, followed by PAV at 100°C (abbr. PAV 100) for 8h, 20h, and 48h
- RTFOT, followed by PAV at 75°C (PAV 75) for 48h, 120h, and 220h
- RCAT at 163°C for 4h, then at 90°C for 17h, 65h, and 140h

PAV, developed within SHRP and standardized under EN 14769 [13], is used to simulate long-term aging in the field. It is carried out after a short-term aging test RTFOT. In this study, PAV tests generally follow the standard. The use of different temperatures and aging times is to study aging kinetics. Aging kinetics is believed to be binder specific and of importance for development of a performance-based binder specification.

RCAT is another long term aging test method newly standardized [14]. It is also claimed that RCAT at 163°C for 4h (normally used as the first step of RCAT) can simulate short term aging [14] [16]. In the test, about 525g of the sample, which has been heated to flow and homogenized, is poured into the cylinder of the device. The short-term aging is performed at 163°C for 4h (RACT163), with air flow rate of 4 L/min and a rotation speed of 5 rev/min. Afterwards, the long-term aging is conducted at 90°C for 140h (RCAT90), with an oxygen flow rate of 4.5 L/h and a rotation speed of 1 rev/min. A test portion of 30g is taken at the end of RCAT163, as well as after 17h and 65h of RCAT90. Aged binder is finally collected after 140h of RCAT90.

2.2 Field samples

Several roads of different ages were selected from the Swedish LTPP database to drill asphalt cores in 2003. The whole asphalt layers of interest (Table 1) were cut from the field asphalt cores for extraction of the binders. Recovery of binder was carried out by a procedure with rotary evaporator (FAS Method 419-02), which in principle is the same as EN 12697-3 [17].
Table 1: Road sections and asphalt materials selected for field aging study

The section E-RV34-1 in Brokind was built in 1969 with a 25 mm MAB12T surface layer on 100-mm macadam. MAB12T is a dense graded asphalt concrete with maximum aggregate size of 12 mm and was produced in 1969 with bitumen A120. The section was overlaid with the same type of asphalt mixture in 1971, and maintained subsequently by other measures as illustrated in Figure 1. The two MAB12T layers were chosen to study field aging after recovery of the binder.

The second road section selected in this study was D-RV53-2 in Nyköping (Figure 1). This section was constructed in 1987 using 40 mm MABT16, a dense graded asphalt concrete with maximum aggregate 16 mm and produced with bitumen B180. The mixture consisted of about 6% (by weight) binder and 3.5% voids. About six years later, the section was overlaid with 28 mm hard dense graded asphalt concrete (HABT).

The third section was D-RV53-1 in Kvicksund, which was built in 1977 with a 25 mm MAB16T. According to the technical requirements of that time [18], MAB16T was a dense graded asphalt mixture with maximum aggregate size of 16 mm and with about 6.2 wt% binder (normally A120) and 3-5% voids. The layer was on the top of the pavement for five years because of a surface treatment in 1982, and several other maintenance measures in the years after (Figure 1).

Test sections in a high traffic road E18/E20 Örebro were also selected to compare laboratory aging and field aging. Those sections were constructed in 1989 with six different types of binder, including penetration grade bitumen B85 and SBS polymer modified binder PMB20. Unfortunately, major part of the test sections was removed due to...
reconstruction, thus field samples cannot be taken. Instead, literature data [19] on the extracted binders after 10-year service were used for comparison.

2.3 Evaluation methods

Rheological characterization of binders was carried out using a dynamic shear rheometer (Rheologia Stress Tech) at two temperatures (25 and 60°C) and at five frequencies ranging from 0.1 to 20 Hz. In all the tests, parallel plates with 25 mm diameter and 1 mm gap were used. The measured parameters included complex modulus $G^*$ and phase angle $\delta$.

Depending on available amount of the samples, penetration and softening point were also measured in many cases.

To chemically evaluate bitumen aging, Fourier transform infrared spectroscopy (FTIR, instrument from Perkin Elmer) was applied to assess carbonyl and sulfoxide functionalities. As products of oxidation reaction, these two functional groups may indicate the degree of oxidative aging in the bitumen. In FTIR analysis, sample solutions of 5% (by weight) were prepared in carbon disulfide. Blank (solvent) and sample scans were performed in a circular sealed cell with KBr windows and 1 mm thickness. The amounts of sulfoxide and carbonyl compounds were assessed by measuring the areas of IR bands at about 1030 and 1705 cm$^{-1}$, respectively.

3. RESULTS AND DISCUSSION

3.1 Laboratory aging

In this study, RTFOT was selected as a short term aging test because of its popularity. Aged samples after RCAT at 163°C for 4h were also collected for comparison since RCAT163 was claimed as one of the options for short term aging before in the long-term aging test by RCAT [14]. Evaluation of aging was carried out by various physical, rheological and chemical property measurements, including penetration, softening point, complex modulus, phase angle, and functional groups (sulfoxides and carbonyls).

Table 2 shows DSR data before and after aging with RTFOT and RCAT163. As indicated by complex modulus and phase angle, the two short-term aging tests are quite similar both for unmodified and SBS polymer modified binders. This is in agreement with observation reported in [16].

For long-term aging during in-service, PAV at 75°C and 100°C and RCAT at 90°C were evaluated. Figure 2 confirms that the temperature is a factor significantly influencing the aging kinetics and the temperature effect is strongly binder-related. For unmodified bitumen, under the tested durations, complex modulus versus aging time can be fitted by a linear regression, both in PAV and RCAT. The general rule that increasing temperature by 10°C doubles the rate of aging is also likely followed in PAV. For example, to reach the same level of complex modulus of 400 kPa at 25°C and 10 rad/s, for bitumen A120, aging time in PAV 100°C is estimated to be 22h, which is just between 1/4 and 1/8 of the time required in PAV 75°C (120h). However, the rule is not valid when PAV and RCAT are compared to each other. The different tests result in different aging kinetics. To obtain a similar level of aging, RCAT will take much longer time than PAV if the temperature is the same. The DSR data, as well as softening point and penetration (Figure 3), show that for the unmodified bitumens, aging of 140h in RCAT at 90°C is more or less equivalent to 20h PAV at 100°C, or 120h PAV at 75°C.

On the other hand, for the polymer modified binder (PMB20), aging kinetics is very different from those of unmodified bitumens. Figure 2 shows that, in PAV 75°C, complex modulus of the modified binder increases gradually with the aging time. When temperature is raised from 75°C to 100°C in PAV or 90°C in RCAT, the rheological property changes become completely unpredictable. This is also the case for softening point change. Unlike unmodified
bitumens, softening points of the SBS modified binder do not increase with the aging time, as illustrated in Figure 4. This is due to a combined effect of bitumen oxidation and polymer degradation (Cf. Section 3.3). Experiments with gel permeation chromatography (GPC) made on a similar modified bitumen (6% SBS) showed that, after RTFOT followed by PAV at 100°C for 20 h or PAV at 75°C for 110 h, GPC peak heights for the SBS polymer were reduced by about 40% and 60%, respectively. The degradation of the polymer apparently compensates for bitumen oxidative hardening. As a consequence, the equivalence between the long term aging tests established for the unmodified bitumens does not exist for PMB (Figure 3). The strong temperature dependence of aging mechanisms and kinetics also makes prediction of PMB aging in the field very difficult. Normally the maximum pavement temperature is about 60°C. Thus the long term aging test conducted a temperature close to 60°C should be the most relevant, in this case it is PAV at 75°C.

Figure 2: Long-term aging tests with PAV and RCAT under different conditions for bitumen A120 and SBS modified binder PMB20 – Rheological measurements by DSR (In PAV, 0 h aging = RTFOT, and in RCAT90, 0 h aging = RCAT 163°C/4h)
Figure 3: Comparison of the long-term aging tests by testing of penetration and softening point

Figure 4: PAV tests under different conditions – Measurements of penetration and softening point (0 h aging in PAV = RTFOT)
3.2 Field aging

In Table 3, the results of complex modulus, penetration and softening point (R&B) measurements are shown for the binders extracted from old pavements. Based on Figures 2 and 4, and by using appropriate curve fitting, we can estimate laboratory aging times required to give the same level of aging as in the field. Figure 5 illustrates examples of estimation for PAV at 100°C. Results estimated for other tests are shown in Table 4.

<table>
<thead>
<tr>
<th>Recovered binders</th>
<th>Sources</th>
<th>Penetration, dmm</th>
<th>R&amp;B, °C</th>
<th>G* at 10 rad/s, kPa</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>at 25°C</td>
</tr>
<tr>
<td>A120</td>
<td>MAB16T, 1977</td>
<td>101</td>
<td>44.0</td>
<td>214</td>
</tr>
<tr>
<td>B180</td>
<td>MABT16, 1987</td>
<td>114</td>
<td>42.7</td>
<td>185</td>
</tr>
<tr>
<td>A120</td>
<td>MAB12T, 1969</td>
<td>36</td>
<td>57.3</td>
<td>1140</td>
</tr>
<tr>
<td>A120</td>
<td>MAB12T, 1971</td>
<td>90</td>
<td>46.8</td>
<td>270</td>
</tr>
<tr>
<td>B85 [19]</td>
<td>ABT, 1989</td>
<td>56</td>
<td>53.5</td>
<td>--</td>
</tr>
<tr>
<td>PMB20 [19]</td>
<td>ABS, 1989</td>
<td>88</td>
<td>78.5</td>
<td>--</td>
</tr>
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</table>

Table 3: Rheological and conventional measurements of recovered binders

<table>
<thead>
<tr>
<th>Recovered binders and sources</th>
<th>Equivalent laboratory aging time (hours) estimated by different parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PAV 100</td>
</tr>
<tr>
<td></td>
<td>Pen</td>
</tr>
<tr>
<td>A120 (MAB12T, 1969)</td>
<td>55</td>
</tr>
<tr>
<td>A120 (MAB12T, 1971)</td>
<td>12</td>
</tr>
<tr>
<td>A120 (MAB16T, 1977)</td>
<td>8</td>
</tr>
<tr>
<td>B85 (ABT, 1989)</td>
<td>4</td>
</tr>
<tr>
<td>PMB20 (ABS, 1989)</td>
<td>2</td>
</tr>
</tbody>
</table>

Table 4: Estimation of equivalent laboratory aging times for the field aged binders

Figure 5: Comparison of field aging and PAV at 100°C
As can be seen from Figure 5 and Table 4, the equivalent aging times for the recovered binders are in general significantly shorter than that currently used or proposed (20h in PAV 100°C and 140h in RCAT 90°C). For example, for the bitumen (A120) extracted from MAB16T in D-RV53-1 (Kvicksund, 1977), only 6-8 hours are required for PAV at 100°C to produce the same level of aging as in the field. If PAV 75°C or RCAT 90°C is used, the equivalent aging time is found to be about 30 hours. It should be noted that this bitumen had been in the pavement for more than 25 years, but only 5 years on the surface. Similar observation of low aging also can be made for the same type of bitumen (A120) that was extracted from MAB12T used in the section E-RV34-1 (Brokind) in 1971.

The relatively low degree of aging is also found for the binder extracted from MABT16 in D-RV53-2 (Nyköping, 1987, Cf. Figure 1). In this section and at the time of drilling asphalt cores, bitumen B180 had been in the road for 16 years, of which 6 years on the surface. However, penetration and softening point of the extracted binder still retain 114 dmm and 43°C, respectively, which are very close to the values after short-term aging with RTFOT (penetration $\approx$ 115 dmm, softening point $\approx$ 42°C). For the corresponding unaged bitumen, penetration is about 180 dmm and softening point about 38°C.

Also for polymer modified binder PMB20, the aging after 10 years of service in the field was very low. Based on penetration and softening point data reported in [19], the equivalent aging time in PAV 100°C is estimated to be only 2-3 hours (Figure 5).

The only exception is observed for bitumen A120 used in Brokind section E-RV34-1, 1969. The extracted binder shows considerable aging. The equivalent aging times in laboratory tests are estimated to exceed 50 hours in PAV 100°C (Figure 5), and to be longer than 400 hours in PAV 75°C or RCAT 90°C (Table 4).

The above observations imply that oxidation of bitumen might have stopped when the bituminous layer was sealed or overlaid. It could also be attributed to low void contents of the asphalt mixtures, which limit access of oxygen into the asphalt layer. For the asphalt cores taken from Kvicksund and Nyköping, void contents were found to be 1.9% and 1.7%, respectively. These values are lower than what had been designed (3-5%). This was probably due to densification under traffic. Because of the low void contents, no effort was made to determine the rate of bitumen aging as function of the depth from the pavement surface. The observed low degree of bitumen aging could also be attributed to relatively low climatic temperature. The recorded average temperature over a year for Brokind and Kvicksund-Nyköping regions was below 10°C.

### 3.3 Chemical aspects - FTIR analysis

Chemical changes during aging have been studied extensively in the past [5] [6] [8]. It is known that oxidation of bitumen produces carbonyls and sulfoxides and increases polarity, causing increases in bitumen viscosity and softening point, etc. In a given aging test, the chemical changes may differ largely between different bitumens, especially between unmodified and polymer modified binders.

Figure 6 shows typical infrared spectrograms for bitumen before aging and after laboratory and field aging. The absorbance band at about 1705 cm$^{-1}$ is attributed to C=O stretch in carbonyl compounds, such as ketones, carboxylic acids and anhydrides. The stretch of S=O in sulfoxides gives an IR absorbance band at around 1030 cm$^{-1}$. By measuring areas of the IR bands, the relative amounts of carbonyl compounds and sulfoxides formed on aging may be assessed.

In Figure 7, IR absorbance ratios of aged to unaged bitumen are plotted against the aging time in PAV; as examples, an unmodified bitumen A120 and SBS polymer modified binder (PMB20) are illustrated. The samples of zero-hour aging in the figure are those after RTFOT. Evidently, at both low (75°C) and high (100°C) PAV temperatures, the formation of sulfoxides is significant. With increasing aging time, the rate of sulfoxides formation tends to decrease and eventually levels off. Unlike sulfoxides, carbonyl compounds are formed at a more constant rate, and the rate is much higher in PAV 100°C than in PAV 75°C. These observations agree with findings reported in the literature [5].
Figure 6: IR spectrograms for bitumen before aging and after laboratory or field aging

Figure 7: IR absorbance ratios as function of aging time in PAV (0 h aging refers to RTFOT)

Figure 7 also indicates that the formation of sulfoxides in the SBS modified binder is lower than that in the unmodified bitumen, while an opposite trend is seen for carbonyl compounds (Note: The base bitumen used for the modified binder has the same source as A120). The inhibiting effect of SBS on sulfoxide formation was also observed in reference [20]. The reason for that is not known, but we suspect that SBS polymer competes with bitumen sulfur compounds (e.g. sulfides) for oxidants. As for carbonyls in the SBS modified binder, the increased amount by aging is contributed not only by bitumen oxidation but also by the degradation of the polymer.

It has been reported that, for a given bitumen, the increase in dynamic viscosity correlates with carbonyls formed on aging [5]. Such relationship does not exist when different bitumens are compared. From this study, similar conclusions can be drawn, as shown by complex modulus in Figure 8. However, for the SBS modified binder (PMB20), correlation between complex modulus and carbonyls is rather poor, which is due to different mechanisms of oxidation in PMB.
Differently from laboratory aging, the field aged bitumens show a much higher level of sulfoxides but lower level of carbonyls, as illustrated in Table 5 and Figure 9. It is likely that higher temperature in laboratory aging results in higher amount of carbonyls while longer time in the field produces higher amount of sulfoxides. These differences imply that oxidation mechanisms of bitumen in the field may not be the same as in laboratory aging test, suggesting difficulty in prediction of field aging by analysis of the functional groups.

Table 5: Relative carbonyl and sulfoxide IR absorbances of bitumen A120 (unaged and aged)

<table>
<thead>
<tr>
<th>IR absorbance</th>
<th>Unaged</th>
<th>RTFOT 100°C, 20h</th>
<th>PAV 100°C, 120 h</th>
<th>Recovered 1969</th>
<th>Recovered 1971</th>
<th>Recovered 1977</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbonyls</td>
<td>0.58</td>
<td>0.91</td>
<td>1.33</td>
<td>1.14</td>
<td>0.80</td>
<td>0.70</td>
</tr>
<tr>
<td>Sulfoxides</td>
<td>0.12</td>
<td>0.12</td>
<td>1.27</td>
<td>1.16</td>
<td>2.51</td>
<td>3.70</td>
</tr>
</tbody>
</table>

Figure 8: Complex modulus as function of IR absorbance of carbonyl compounds formed in PAV tests

Figure 9: Comparison of field aging and laboratory aging based on IR absorbance ratios
4. CONCLUSIONS

From the results presented in this paper, the following conclusions can be drawn:

- Binders from roads of long time in-service generally display a low degree of age-hardening. Based on the rheological and conventional measurements, the estimated equivalent laboratory aging durations are much shorter than those being standardized;
- Low voids in the asphalt mixture and surface sealing and overlay can prevent aging of the binder;
- Aging kinetics and formation of sulfoxides and carbonyls are strongly temperature dependent. It is shown that the increase in bitumen stiffness correlates well with carbonyls formed on aging. For the SBS modified binder, the polymer is found to inhibit the formation of sulfoxides on aging;
- Compared to laboratory aging, much higher level of sulfoxides but lower level of carbonyls is found for the binders aged in the field. This suggests that oxidation mechanisms in the field may not be the same as in laboratory aging tests;
- The short-term aging tests RCAT (163°C, 4h) and RTFOT are quite similar;
- The long-term aging tests PAV (100°C, 20h), PAV (75°C, 120h) and RCAT (90°C, 140h) are almost equivalent for unmodified bitumen; but they are different when applied to the SBS modified binder.

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