Research on Vegetable Oil Based Rejuvenating Agent and Properties of Recycled Asphalt

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Abstract. Based on theory of dissolution and dispersion and special chemical structure of vegetable oil, the article chooses four kinds of vegetable oil (Tung oil, Castor oil, Rapeseed oil and Linseed oil) and blended them with asphalt. And then Rejuvenating Agents of asphalt based on the four kinds of vegetable oil are prepared. The test results show that good exponent relationship is presented between Tung oil content and Brookfield viscosity of Tung-oil-based Rejuvenating Agent. Furthermore, this paper studies the performance of recycled asphalt by conventional indexes, adhesion, SHRP indexes. The data show it by improving the composition of aging asphalt, thereby enabling recycled asphalt indexes recovered to close to or above the level of original asphalt.

Keywords. Vegetable oil, rejuvenating agent, recycled asphalt, properties.

1. Introduction
During storage, mixing, transport and road paving, the asphalt will become aged and lead to pavement failure. These problems cause the expensive preservation of pavements. Recycling asphalt pavement (RAP), as an economic and environmental solution, is applied for waste asphalt mixtures [1]. Moreover, some studies illustrate that using a rejuvenating agent in the aged asphalt mixtures can increase the higher application percentage of the RAP in the pavement [2].

The aging mechanism of asphalt causes loss of volatiles and oxides, which leads to that asphalt, with stiffer and higher viscosity. According to the aging condition of asphalt [3], the Rejuvenating Agent or new asphalt, or both of them are used for Recycling. Rejuvenating Agents restore the original ratio of asphaltenes to maltenes in aged bitumen binders in order to soften the aged binder and create a broad-spectrum rejuvenation that replenishes the volatiles and dispersing oils while promoting adhesion [4]. Currently, Rejuvenating Agents on the market are mostly mixtures of lubrication oil, mineral oil and asphalt, and the adopted materials are mostly series of products made by petroleum [5]. However, it is extremely urgent for developing new Rejuvenating Agents of asphalt due to increasing shortage or even exhaustion of petroleum resource.
The investigations illustrate that the main ingredient of vegetable oil is triglyceride which contains polar ester base and non-polar linear paraffin [6], while the polar groups can absorb asphalts and the non-polar linear paraffin can mutually peptize with melted asphalt to increase compatibility of asphalt and saturates in asphalt when vegetable oil is used as medium (interface agent). Therefore, it can better stabilize the colloid structure of asphalt with the action of load. Moreover, vegetable oil is also a kind of good viscosity modifier with high boiling point and low volatility; In liquid state at normal temperature, it has low viscosity to rapidly dissolve and penetrates in aged asphalt.

Based on solubilisation-dispersion theory [7], the article choose four kinds of common vegetable oils and blended them with BinZhou 110° asphalt, and then Rejuvenating Agents of asphalt based on four kinds of vegetable oil were prepared. The properties of the recycling asphalt are test including penetration, softening point, Brookfield viscosity, rheological properties and Adhesion properties test. The aim of this research is to determine the potential using of vegetable oil as a rejuvenating agent for aged bitumen binder in order to reduce the using of petroleum resources and the expense of highway renovation.

2. Experiment

2.1. Materials

Qinhuangdao 70° base asphalt was used as raw material for preparing aged asphalt. BinZhou 110° asphalt was obtained from the BinZhou refinery in Shan Dong province, China. The base properties of Qinhuangdao 70° asphalt and BinZhou 110° were shown in table 1.

| Name              | 10℃ Ductility(cm) | 15℃ Ductility(cm) | Penetration(dmm) | Softening Point(℃) |
|-------------------|-------------------|-------------------|------------------|--------------------|
| Qinhuangdao 70°   | >100              | >100              | 69               | 51.0               |
| TFOT 70°          | 7.7               | 34.0              | 44               | 57.3               |
| BinZhou 110°      | >100              | >100              | 108              | 43.2               |

This article chose four kinds of vegetable oils, namely Tung oil, Castor oil, Rapeseed oil and Linseed oil. The components of four kinds of vegetable oils were presented in table 2.

| Name         | Saturates (%) | Aromatics (%) | Colloid (%) | Asphalt (%) |
|--------------|---------------|---------------|-------------|-------------|
| Tung oil     | 34.3          | 13.3          | 52.4        | 0.0         |
| Castor oil   | 26.6          | 14.6          | 58.8        | 0.0         |
| Rapeseed oil | 32.0          | 16.5          | 51.5        | 0.0         |
| Linseed oil  | 33.5          | 17.5          | 49.0        | 0.0         |

2.2. Preparation of the Samples

2.2.1. Preparation of Samples for Aged Asphalt. Thin-film oven is used for accelerating the aging of asphalt. The aged asphalt samples are obtained by placing the Qinhuangdao 70° sample in the thin-film oven and keeping the temperature at 163℃ for 5h. The properties of the aged 70° asphalt are listed in table1.

2.2.2. Preparation Process of Rejuvenating Agent. Firstly, binzhou 110° asphalt is heated to the melting state, and keeps the temperature at about 140℃; then add measured vegetable oil and
mechanically mix the both to uniform state to obtain vegetable-oil-based Rejuvenating Agent of asphalt, where the speed of mixer should be 500-800 rpm, and the mixing time should be 20 min. In this paper, TY-3 represents Tung oil Rejuvenating Agent, where m (Tung oil): m (110° Asphalt) = 30: 100, BM-3 represents Castor oil Rejuvenating Agent, where m (Castor oil): m (110° Asphalt) = 30: 100, CZ-3 represents Rapeseed oil Rejuvenating Agent, where m (Rapeseed oil): m (110° Asphalt) = 30: 100, YM-3 represents Linseed oil Rejuvenating Agent, where m (Linseed oil): m (110° Asphalt) = 30: 100.

2.2.3. Preparation Process of Recycled Asphalt. The vegetable-oil-based Rejuvenating Agent of asphalt and aged asphalt sample are heated to 150°C, and then uniformly mix the both. The speed of mixer should be 800-1000rpm, where the ratio for m (Rejuvenating Agent): m (TFOT 70° Asphalt) =10:100. T-3 represents Tung oil Recycled asphalt, where m (TY-3): m (TFOT 70° Asphalt) = 10: 100, B-3 represents Castor oil Recycled asphalt, where m (BM-3): m (TFOT 70° Asphalt) = 10: 100, C-3 represents Rapeseed oil Recycled asphalt, where m (CZ-3): m (TFOT 70° Asphalt) = 10: 100, Y-3 represents Linseed oil Recycled asphalt, where m (YM-3): m (TFOT 70° Asphalt) = 10: 100.

2.3. Performance Measurements

2.3.1. General Performance. Conventional performance is mainly composed of softening point, penetration, ductility, Brookfield viscosity, flash point, thin-film oven (TFOT) and water boiling method according to Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering (JTG E20-2011) [8].

2.3.2. Rheological Property of Recycled Asphalt. Tests for DSR and BBR are done as SHRP specification.

2.3.3. Asphalt Adhesion. Since it is impossible to quantitatively analyse the adhesion difference between asphalt and aggregate by water boiling method, this article also adopts UV spectrophotometry during the test to quantitatively analyse the differences in adhesion between asphalts and aggregates. UV spectrophotometry produced by SHIMADZU are used to conduct Ultraviolet-adhesion test for 70° asphalt and Recycled asphalt (T-3, B-3, C-3 and Y-3) respectively.

3. Results and Discussion

3.1. Preparation of Vegetable-Oil-Based Rejuvenating Agent of Asphalt

| Item                     | TY-1   | TY-2   | TY-3   | TY-4   | TY-5   |
|--------------------------|--------|--------|--------|--------|--------|
| Amount wt (%) 110° Asphalt | 100    | 100    | 100    | 100    | 100    |
| Tung Oil                | 10     | 20     | 30     | 15     | 25     |
| 60°C Brookfield viscosity (mPa•s) | 19780  | 7264   | 3433   | 11410  | 4561   |
| Flash point (°C)        | >270   | >270   | >270   | -      | -      |
| Mass change (%)         | -0.04  | -0.02  | +0.01  | -      | -      |
| Viscosity ratio(after and before TFOT) | 1.96   | 2.28   | 2.71   | -      | -      |

3.1.1. Effect of Vegetable Oil on Properties of Rejuvenating Agent. This article chooses Tung oil, one of the vegetable oils mentioned above, as the example for preparing vegetable-oil-based Rejuvenating Agent of asphalt to examine the influence of vegetable oil content to the Rejuvenating Agent of asphalt. They are named as TY-1 to TY-5 according to different Tung oil content in the Rejuvenating
Agent. The effects of Tung oil on properties of Rejuvenating Agent of asphalt were illustrated in table 3.

Since the viscosity of Tung oil at the same temperature is significantly smaller than that of 110# asphalt, Brookfield viscosity of Rejuvenating Agent becomes smaller continuously with the increase of Tung oil amount. Tung oil contains less flammable substances and very high boiling point and burning point. Therefore, Rejuvenating Agent also has high flash point. The mass change of Tung oil Rejuvenating Agent tends to rise with the increase of Tung oil amount after short-term aging. The main cause is that main content of Tung oil is Tung oil glyceride whose segments contains highly reactive conjugated double bond and conjugated triple bond that has significantly higher reactivity than that of asphalt. It greatly increases the opportunity for reaction of active components in the Rejuvenating Agent and oxygen to result in rise trend for mass trend of Rejuvenating Agent. The mass change becomes positive when the Tung oil amount comes to a certain level. In the process of film oven heating, the light components in the regeneration agent volatilize, the active component "oxygen absorption", the fact that tung oil is a dry oil and self-polymerization will occur under high temperature conditions, generating tung oil based polymer macromolecules, resulting in film oven heating viscosity ratio increases with the growth of the content of tung oil in the regeneration agent.

The curve of 100 times the Tung oil amount versus Brookfield viscosity of Rejuvenating Agent is plotted, as showed in figure 1. It can be found that there is a good exponent relationship between Brookfield viscosity of Rejuvenating Agent and amount of Tung oil, where the detailed relationship is $Y=85751X^{1.61}$ and correlation coefficient is $R^2=0.9938$. This conclusion can be used as the basis of different viscosity grade for preparing the Rejuvenating Agent.

![Figure 1. Curve of Recycling agent Brookfield Viscosity versus amount of Tung Oil.](image)

### 3.1.2. Effect of Vegetable Oil Species versus Properties of Rejuvenating Agent

Another three kinds of vegetable oils, namely Castor oil, Rapeseed oil and Linseed oil, are chosen to prepare BM-3, CZ-3 and YM-3 Rejuvenating Agent s of asphalt, based on amount of Tung oil (30%) determined by TY-3 Rejuvenating Agent. The effect of vegetable oil species versus the property of Rejuvenating Agent is shown in table 4.

| Item                   | TY-3 | BM-3 | CZ-3 | YM-4 |
|------------------------|------|------|------|------|
| 60°C Brookfield viscosity (mPa*s) | 3433 | 4600 | 1860 | 1640 |
| Flash point (°C)       | >270 | >270 | >270 | >270 |
| Mass change (%)        | +0.01| -0.03| -0.05| -0.01|
| Viscosity ratio        | 2.71 | 1.36 | 1.72 | 1.69 |

The different vegetable oils have great effect on Brookfield viscosity of Rejuvenating Agent, where BM-3 has the maximum Brookfield viscosity and YM-3 has the minimum Brookfield viscosity mainly
because of different apparent viscosity value of each vegetable oil at the same temperature. All the four Rejuvenating Agents have high flash points that exceed 270°C. The mass change of the four Rejuvenating Agents are not obvious before and after heating in the thin-film oven but relatively small which means that the anti-aging property is excellent. There is no significant difference in the quality of the four regenerants before and after heating in the film oven, but they have one thing in common: they were relatively small, indicating that they had excellent anti-aging properties. After heating, the film oven of the four reagents have a large viscosity ratio difference, among which TY-3 was the largest and BM-3 was the smallest. The main reason is that tung oil contains highly active double and triple bonds, which could react with oxygen at high temperature. Therefore, TY-3, the regenerator, is more serious in "oxygen absorption" during the aging process. The main content in Castor oil of CZ-3 is trime ricin oleic acid glyceride that belongs to non-drying oil with less active bonds than the other three kinds of vegetable oils and highest oxidation stability. Therefore, the viscosity of Castor oil Rejuvenating Agent is small after aging in thin-film oven.

3.2. Research on Properties of Recycled Asphalt.

3.2.1. General Properties of Recycled Asphalt. The above prepared TY-3, BM-3, CZ-3 and YM-3 vegetable oil Rejuvenating Agents are added into aged asphalts respectively to prepare Recycled asphalts, named as T-3, B-3, C-3, and Y-3 respectively. The general properties of Recycled asphalt are showed in table 5.

| Item                | T-3  | B-3  | C-3  | Y-3  | 70\# |
|---------------------|------|------|------|------|------|
| 135°C Brookfield viscosity (mPa•s) | 560.5 | 563.5 | 538.1 | 550.6 | 428.5 |
| 25°C Penetration (dmm)        | 65   | 68   | 67   | 71   | 69   |
| Softening point (°C)          | 52.4 | 52.5 | 51.9 | 50.3 | 51.0 |
| 10°C Ductility (cm)           | 26.5 | 26.9 | 32.7 | 40.3 | >100 |
| 15°C Ductility (cm)           | >100 | >100 | >100 | >100 | >100 |
| TFOT Mass change (%)          | -0.40 | -0.47 | -0.35 | -0.40 | -0.46 |
| 10°C Ductility (cm)           | 7.3  | 8.1  | 7.9  | 8.0  | 7.7  |
| Penetration ratio (%)         | 69.2 | 66.2 | 70.1 | 64.8 | 63.8 |

After adding the four vegetable oil Rejuvenating Agents, the Rejuvenating Agents, including large amount of low molecular weight components in the vegetable oil, it can dissolve and penetrate into the aged asphalt to reduce difference of solubility parameter for asphalt and melted asphalt and form a stable colloid solution. Compared with the aged 70\# asphalt, the Recycled asphalt have the increasing of penetration and ductility as well as reducing of softening point and Brookfield viscosity. And the ductility (10°C) of the Recycled asphalt obviously lower than that of original sample. After being heated in thin-film oven for aging, the Recycled asphalt (except for B-3) has smaller mass change than that of 70\# asphalt because of slight volatilization of the Rejuvenating Agent after TFOT. All the Recycled asphalt (except for T-3) has slightly larger residual ductility than that of 70\# asphalt. It means that the Rejuvenating Agent contains great amount of plasticizing ingredient which results in god plastic deformation capacity of the Rejuvenating Agent, and the vegetable oil can keep its stable property before and after subject to short term aging. There are two main reasons: One is the excellent heat resistance property of vegetable oil, and the other is mutual dissolving and dispersing of vegetable oil and asphalt to mutually protect each other. The residual penetration of the four Recycled asphalts is a little higher than that of 70\# asphalt mainly because of high anti-aging property of Rejuvenating Agent and reduction of re-aging of 70\# asphalt after aging.
3.2.2. Adhesion of Recycled Asphalt. The adhesion between asphalt and aggregate is different because of polarities of four components in asphalt. Saturates mainly participate in weak physical action with the aggregate and are apt to peel from the surface of aggregate; while colloid and asphalt are strongest polarity components, therefore, the adhesion of the asphalt with more colloid and asphalt are also stronger. For 70° asphalt, the asphalt content increases after aging, which results in strengthening of adhesion. After adding Rejuvenating Agent in aged 70° asphalt, the percentage of asphalt content begins to reduce so as to result in reducing of adhesion for Recycled asphalt compared with aged asphalt in table 6. The vegetable oil contains large amount of C=O genes that can result in increasing of hydrophilic group content in vegetable oil Rejuvenating Agent. When the force between aggregate and polar group is smaller than the force between polar group and water, the asphalt will peel away from the aggregate surface and results in reducing of adhesion. Adhesion of B-3 Recycled asphalt is slightly worse than that of 70° asphalt, because Castor oil contains not only polar bond C=O but also large amount of hydrophilic groups –OH which will have hydrogen bonds action with hydrone. When the action of hydrogen bonds is larger than the absorption action, the peeling of asphalt from aggregate surface will be further exacerbated and results in worse adhesion of Castor-oil-based Recycled asphalt.

| Item | 70° | T-3 | B-3 | C-3 | Y-3 | TFOT 70° |
|------|-----|-----|-----|-----|-----|----------|
| $A^\alpha$ | 0.266 | 0.282 | 0.243 | 0.265 | 0.238 | 0.296 |
| $A^\beta$ | 0.256 | 0.278 | 0.235 | 0.261 | 0.231 | 0.290 |
| $A^\gamma$ | 0.262 | 0.280 | 0.240 | 0.263 | 0.235 | 0.292 |
| $R_S$ (%) | 60.8 | 58.0 | 63.3 | 51.0 | 51.0 | 34.7 |

3.2.3. Rheological Property of Recycled Asphalt. Asphalt, as viscoelastic material, displays different characteristics of application properties in paving industry. Thus, it is crucial to examine the rheological properties of asphalt. In order to evaluate the influence of Rejuvenating Agent on rheological properties of asphalt, all above Recycled asphalts are prepared. In the same way, procedure and the rheological tests are essentially non-destructive, differences in their rheological behavior are attributed exclusively to their composition and molecular structure of the Rejuvenating Agent. The rheological behavior of the Recycled asphalts is summarized in figures 2-4, comparing with that of 70° asphalt.

Figure 2. Curve of $G*/\sin(\delta)$ versus Temperature.
Figure 3. Curve of Creep Stiffness versus Temperature on Different Recycling Asphalts.

Figure 4. Curve of Creep Rate versus Temperature on Different Recycling Asphalt.

1) Dynamic shear rheology test (DSR). It can be known from figure 3 that the reducing rate of $G'/\sin\delta$ becomes faster with the increase of temperature. The higher the temperature rises, the worse the ability resists deformation due to external force of the four Recycled asphalts becomes. The value of $G'/\sin\delta$ for the four kinds of vegetable-oil-based Recycled asphalt are all higher than that of original 70° asphalt at the same temperature. However, the result obtained by DSR is a little inconsistent with the softening point results tested above. There is a good correlation between $G'/\sin\delta$ in SHRP that reflects high temperature stability and equivalent softening point $T_{800}[9]$.

The penetration index and equivalent softening point $T_{800}$ of different Recycled asphalts are list in table 7. Based on high temperature results given for DSR, $T_{800}$, and softening point, the priorities will be B-3>T-3>C-3>Y-3>70°; B-3>T-3>C-3>Y-3>70°; and B-3>T-3>C-3>70°>Y-3. For priorities of high temperature properties reflected by different indicators, $G'/\sin\delta$ is consistent with $T_{800}$ but is inconsistent with softening point. The main reason is that domestic asphalt has high wax content, and temperature rise speed, mixing speed etc. may affect the test result of softening point.

Table 7. Penetration Index and Equivalent Softening Point $T_{800}$ of Different Recycling Asphalts.

| Item    | Penetration / dmm | Fitting curve | PI     | $T_{800}$ |
|---------|-------------------|---------------|--------|-----------|
|         | 15°C 25°C 30°C    | Slope A       | Intercept K |         |
| 70°     | 24 69 116         | 0.04565       | 0.69596 | -0.86     | 48.94     |
| TFOT 70° | 19 44 74          | 0.03895       | 0.68828 | 0.18      | -         |
| T-3     | 24 65 101         | 0.04184       | 0.7561  | -0.3      | 51.96     |
| B-3     | 27 68 109         | 0.04036       | 0.8253  | -0.06     | 52.15     |
| C-3     | 25 67 109         | 0.04266       | 0.75845 | -0.42     | 50.91     |
| Y-3     | 26 71 114         | 0.04291       | 0.77304 | -0.46     | 50.27     |

2) Flexural creep stiffness test (BBR). To better reflect the low temperature property of asphalt, this article adopts BBR to test the low temperature rheology property of Recycled asphalt and compare the results with that of 70° asphalt. Concrete contents are in figure 3 for creep stiffness and figure 4 for creep rate obtained from BBR. It can be seen from figure 3 that the creep stiffness modulus of the five kinds of asphalt increases gradually with the decrease of temperature. When comparing the -12°C to -18°C region with the -18°C to -24°C region, the value of S increases sharply to indicate the sharp strengthening of brittleness of asphalt. All the five kinds of asphalts meet the requirement of $S\leq300$MPa at -18°C. Best low temperature cracking resistance property can be obtained when S value is the minimum at the same temperature. This article arranges the low temperature property of the asphalts according to S value at -18°C with the priorities as follows: Y-3>C-3>T-3>B-3>70°.
Meanwhile, it can be known from figure 4 that the m value for the five kinds of asphalt samples are all larger than 0.3 when the temperature is higher than -24°C; the m value will continuously increase with the increase of temperature, i.e. the cracking resistance property of asphalt is better when temperature is higher. All the five kinds of asphalts have excellent cracking resistance property since the m value represents stress relaxation of asphalt. It should be noticed for C3 Recycled asphalt that the m value changes slightly with the decrease of temperature when the temperature is within the range of -12°C to -18°C; however, the m value decreases obviously when the temperature decreases to -24°C. The m value of Y3 Recycled asphalt has the most smooth change with temperature change in the five kinds of asphalt samples. The essential reason for the above phenomenon is that: on the one hand, the apparent viscosity changes are also different, resulting in different thermal motion rates and internal friction forces between molecules; on the other hand, the liquid-solid phase transition temperatures of each vegetable oil are also different, so the interaction forces between the molecules are various at different temperature intervals.

The creep stiffness for the five kinds of asphalt samples conforms to the specification requirement at -18°C in SHRP. In this article, -18°C is taken as the lowest temperature limit of asphalt to compare the values of m. larger value of m will correspond to better stress relaxation property of asphalt and better cracking resistance property. The priorities is C3 > Y3 > T3 > B3 > 70#.

From figure 3, low temperature property of vegetable-oil-based Recycled asphalt is better than that of fresh 70# asphalt. The reason is that vegetable oil contains large amount of long-chain fatty acid substances that results in higher flexibility of asphalt. Therefore, vegetable-oil-based Recycled asphalt has lower flexural stiffness modulus and better corresponding low temperature cracking resistance properties at the same temperature.

4. Conclusions
The main purpose is to investigate the effects of Vegetable-oil rejuvenating agent for aged bituminous pavement by assessing the physical properties of the rejuvenating agent and recycling asphalts. From these preliminary results, the following conclusions can be drawn.

1) Tung oil, Castor oil, Rapeseed oil and Linseed oil have “Amphiphilic Structure”, which has excellent dissolving and dispersing abilities to asphaltene. They are excellent raw materials for preparing Rejuvenating Agents. And the Vegetable-oil rejuvenating agent is an antioxidant or rejuvenator.

2) When m (vegetable oil): m (110# asphalt) = 30:100 and the amount of prepared vegetable-oil Rejuvenating Agent is 10% of the mount of aged asphalt, the prepared recycling base asphalt can reach to or approximate to the level of fresh 70# asphalt with regard to common indicators. The higher the vegetable oil content is, the lower the softening point and the higher the ductility and penetration of prepared Recycled asphalt will be.

3) UV spectrophotometry are adopted to quantitatively represent the adhesion property. It is indicated that the adhesion between asphalt and aggregate is improved after aging of the asphalt, but the adhesion decreases after adding Rejuvenating Agent due to percentage decrease of asphalt.

4) It is indicated form DSR test that Recycled asphalt contains more elastic components than that of fresh 70# asphalt, therefore the high temperature stability of Recycled asphalt is higher than that of fresh 70# asphalt; it is also obtained from DSR test that low temperature cracking resistance of vegetable oil Recycled asphalt is better than that of fresh 70# asphalt, which indicates that vegetable oil can better recover the low temperature property of aged asphalt.

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