Prediction of Rheological Properties of Bio-asphalt Binders Through Response Surface Methodology

A M Al-Sabaeei1*, M Napiah1, M Sutanto1, W Alaloul1, N I M Yusoff2 and A A S Ghaleb1

1Department of Civil & Environmental Engineering, Universiti Teknologi PETRONAS, 32610 Bandar Seri Iskandar, Perak, Malaysia.
2Department of Civil and Structural Engineering, Universiti Kebangsaan Malaysia, 43600, Bangi, Selangor, Malaysia
*Corresponding author: abdulnaser_17005477@utp.edu.my

Abstract. In this study, the response surface methodology (RSM) technique was applied to model the high-temperature rheological properties of bio-asphalt binders, incorporating crude palm oil (CPO) under unaged and short-term ageing conditions. The interaction effects of CPO at 0%, 5%, 10% and 15% by weight of the base asphalt binder and test temperatures of 40 °C–76 °C as independent variables on the complex modulus and phase angle were evaluated. Results show that CPO content and test temperature have significant effects on the complex modulus and phase angle of bio-asphalt binders. RSM statistical analyses present high degrees of correlation ($R^2$) of 0.9980, 0.9986, 0.9820 and 0.9682 for the dependent variables' complex modulus and phase angle under unaged and aged conditions, indicating that the developed models are consistent with the experimental results. The numerical optimisation shows that the optimum complex modulus and phase angle of bio-asphalt binders can be achieved with 5% CPO at a temperature of 64 °C.

1. Introduction
The demand for bitumen has increased rapidly over the past 10 years with the rapid increase of highways and airport pavement construction and maintenance. However, the conventional petroleum-based binder is produced from nonrenewable resources, which involve crude oil refining. Therefore, the need for substitute materials for conventional asphalt binders that can reduce the high dependency of bitumen and decrease the pavement construction cost is urgent [1-2]. Incorporating further biomaterials in pavement engineering application is the first step. Several studies have revealed that biooils derived from renewable resources show similar viscoelasticity and chemical combination as those of asphalt binder, which can be utilised as a partial replacement. Furthermore, most of the bio-oils added in asphalt binders reportedly enhance the low and intermediate temperature resistances, although unfortunately reduce the rutting resistance of bio-modified binders [3-4]. Although several researchers have studied bio-asphalt binders, conclusive outcomes have yet been established; nevertheless, a wide agreement exists on the utilisation of bio-oils as a potential additive in petroleum-based asphalt binders [3, 5]. Therefore, the need to evaluate the bio-oils from different biomass resources as a partial replacement in asphalt binders has been expressed, especially those with properties similar to asphalt binders that more likely simultaneously enhance the rutting, fatigue and low-temperature resistance [4, 6].

Crude palm oil (CPO) is a substantial raw biomaterial for the manufacturing of food and nonfood products, such as cooking oil, biodiesel and cosmetics and contributes 30% to the international oil
production [7-8]. Among the various bio-oils that have been utilised as an additive in petroleum-based asphalt binders, limited research has incorporated CPO despite its worldwide availability. It was reported that 64.5 million metric tons of the palm oil was produced in 2016 and 2017 [9]. Furthermore, 80% of the overall palm oil in the world is produced by Malaysia and Indonesia. CPO contributes to the highest quantity in the international market compared to other vegetable oils [10]. CPO has a higher oxidation resistance and a lower water content than most biomaterials [11]. Therefore, CPO is one of the strongest candidates for investigation among different bio-oils as a bio-asphalt for green and sustainable pavement applications. Several models have been developed for prediction the rheological characteristics of asphalt binders. However, few studies have been carried out to model the rheological properties of bio-binders. Several published works have applied response surface methodology (RSM) in pavement material modelling for its excellent capability of representing the correlation among dependent and independent variables [12-14].

RSM is a statistical and mathematical approach that uses numerical and graphic representations for establishing models among responses and one or more independent variables [15]. RSM is mainly utilised for experimental design, modelling and optimisation at few experimental runs [12, 16]. RSM has been utilised in several research areas, such as manufacturing engineering, biomaterials science and concrete materials. Currently, The application of RSM in pavement engineering has been increasingly recognised [14]. The present study aims to develop models to predict the rheological properties of CPO-modified bitumen in terms of linear viscoelastic properties under unaged and short-term aging conditions.

2. Materials and method

2.1. Materials

Asphalt binder with 60/70 penetration grade, which was supplied by PETRONAS Refinery, Malacca, Malaysia was utilised in this study. Its physical and rheological characteristics were tested to ensure that they satisfy the Malaysian requirements. The CPO used in this study was supplied by Flecra Processing and Engineering Sdn. Bhd., Perak Malaysia. It has a softening point of 33 °C–40 °C and density of 0.899–0.920 g/cm³ at 40 °C. The CPO was filtered for any impurities before usage. The CPO concentrations applied to produce the bio-asphalt binders were 0%, 5%, 10% and 15% by weight of bitumen.

2.2. Methods

2.2.1. Preparing of bio-asphalt samples.

As previously mentioned, the CPO-modified binders (bio-asphalt binders) were prepared by addition of different CPO concentrations, as recommended by the RSM software. The base asphalt binder was heated in an oven for one hour at 160 °C to ensure that its fluidity is sufficient for mixing. Homogenous blends of base asphalt binder and CPO were achieved at a shear rate of 1000 rpm and temperature of 140 °C for 1 h. Finally, the blends were carefully stored prior to testing for rheological properties.

2.2.2. Short-term aging test.

The short-term aging test was carried out by the rolling thin film oven (RTFO) according to ASTM D2872 2012 [17] to simulate the ageing of bitumen during the production and applied of the asphalt mixture in road construction. Eight samples of 35 g each were poured into the RTFO containers. Then, the containers with specimens were placed in a RTFO carriage at 163 °C. The ageing process was performed for 85 min at 15 rpm carriage rotation with air flow into each glass at 4000 mL/min. The residual from the glass was transferred for the aged rheological property tests.

2.2.3. Dynamic shear rheometer test.

The viscoelastic properties of the base and bio-binders bitumen were obtained utilising a Kinexus Pro+ dynamic shear rheometer based on ASTM D7175-15 [18]. Temperature sweep test was conducted at a standard frequency of 1.59 Hz under a control strain loading mode. The test was carried out at a
temperature range of 40 °C–76 °C with an increase of 2 °C/min and 25 mm geometry diameter with a 1 mm gap according to the standard requirements. The complex modulus ($G^*$), phase angle ($\delta$), rutting factor ($G^*/\sin\delta$) and fatigue factor ($G^*\times\sin\delta$) were determined under control and aged conditions for the base and bio-asphalt binders at different CPO concentrations.

2.2.4. Design of experiment and method of analysis using RSM.

The Design Expert software 10.0.08 was utilised to carry out the RSM. The user-defined option was applied to establish the mathematical model and statistical analysis of the CPO content at various test temperatures. The user-defined option was selected according to the available data and the number of variables levels, which mostly for the experimental data has been already collected, user-defined is recommended for developing and analyse the model [19-20]. The variables considered in the RSM are as follows: CPO content and temperatures of the test at seven levels (i.e. 40, 46, 52, 58, 64, 70 and 76 °C). A total of 28 experiments were performed to evaluate the $G^*$ and $\delta$ at control and aged conditions. The experimental design matrix and the dependent variables (responses) are presented in Table 1.

**Table 1.** Experimental design layout

| Run | Factor | Response | A: CPO (%) | B: Temp. (°C) | Complex modulus (kPa) | Phase angle (°C) |
|-----|--------|----------|------------|--------------|----------------------|----------------|
|     |        |          | Unaged     | Aged         | Unaged               | Aged            |
| 1   | 5      | 70       | 0.51       | 1.26         | 85.41                | 83.08           |
| 2   | 15     | 52       | 0.63       | 1.06         | 86.2                 | 84.28           |
| 3   | 5      | 46       | 13.53      | 29.30        | 76.59                | 70.99           |
| 4   | 0      | 58       | 2.42       | 10.20        | 82.61                | 81.74           |
| 5   | 10     | 58       | 0.69       | 1.30         | 86.82                | 84.8            |
| 6   | 15     | 58       | 0.30       | 0.49         | 87.42                | 86.12           |
| 7   | 10     | 64       | 0.33       | 0.62         | 87.88                | 86.32           |
| 8   | 10     | 70       | 0.17       | 0.31         | 88.57                | 87.4            |
| 9   | 5      | 40       | 33.19      | 56.70        | 73.13                | 68.11           |
| 10  | 10     | 52       | 1.51       | 2.84         | 85.39                | 82.63           |
| 11  | 0      | 46       | 14.36      | 45.60        | 77.21                | 77.05           |
| 12  | 15     | 40       | 3.40       | 6.66         | 82.38                | 78.24           |
| 13  | 5      | 64       | 1.03       | 2.63         | 84.18                | 80.76           |
| 14  | 10     | 76       | 0.09       | 0.17         | 88.9                 | 88.02           |
| 15  | 10     | 46       | 3.64       | 6.12         | 83.45                | 80.18           |
| 16  | 0      | 64       | 1.03       | 3.23         | 84.57                | 84.68           |
| 17  | 0      | 70       | 0.47       | 1.46         | 85.91                | 86.18           |
| 18  | 5      | 52       | 5.36       | 13.20        | 79.81                | 74.5            |
| 19  | 0      | 76       | 0.13       | 0.69         | 86.74                | 87.29           |
| 20  | 5      | 58       | 2.27       | 5.82         | 82.32                | 77.87           |
| 21  | 0      | 52       | 6.05       | 18.80        | 79.95                | 79.91           |
| 22  | 0      | 40       | 29.82      | 98.30        | 74.97                | 74.46           |
| 23  | 15     | 46       | 1.38       | 2.48         | 84.64                | 81.74           |
| 24  | 5      | 76       | 0.25       | 0.62         | 86.18                | 84.87           |
| 25  | 15     | 70       | 0.09       | 0.13         | 88.65                | 88.12           |
| 26  | 15     | 64       | 0.16       | 0.24         | 88.17                | 87.34           |
After verifying that the linear model cannot represent the data in this study, the appropriate higher-order polynomial model was utilized to represent the curvature correlation between the independent and responses and optimise the prediction condition. The second-order function recommended in [20] was applied as shown as follows:

\[ y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i<j}^{k} \beta_{ij} X_i X_j + \varepsilon, \]  

(1)

where \( y \) is the predicted dependent variable, \( \beta_0 \) is the \( y \)-intercept at \( X_1 = X_2 = 0 \), and \( k \) is the number of factors utilised in the analysis. \( X_i \) and \( X_j \) are the coded values of independent variables, where \( i \) and \( j \) are the coefficient for the linear and quadratic equations, respectively. \( \varepsilon \) denotes the error.

3. Results and discussions

3.1. Statistical and ANOVA analyses

Statistical analyses were performed to have an adequate understanding of the performance of the models in terms of \( G^* \) and \( \delta \). Firstly, regression analyses were performed. Then, fitted cubic models were developed to predict the \( G^* \) and \( \delta \). The selection of the cubic model was based on the RSM recommendation to avoid any aliases by the software. Equations 2–5 show the developed model equations for \( G^* \) and \( \delta \) under unaged and aged conditions.

\[
\text{Ln (Complex modulus – unaged)} = 8.40835 + 0.38259 \times A - 0.11028 \times B - 7.76208E - 003 \times A \times B - 0.055935 \times A^2 - 3.29960E - 004 \times B^2 - 6.60607E - 005 \times A^2 \times B + 9.32712E - 005 \times A \times B^2 + 2.26080E - 003 \times A^3
\]  

(2)

\[
\text{Ln (Complex modulus – aged)} = 9.30816 + 0.19958 \times A - 0.10603 \times B - 5.29937E - 003 \times A \times B - 0.047499 \times A^2 - 2.95728E - 004 \times B^2 - 1.68609E - 004 \times A^2 \times B + 7.65868E - 005 \times A \times B^2 + 2.30820E - 003 \times A^3
\]  

(3)

\[
\text{Phase angle (Unaged)} = 41.48255 - 0.59985 \times A + 1.04622 \times B - 0.013223 \times A \times B + 0.31637 \times A^2 - 5.83085E - 003 \times B^2 - 0.013564 \times A^3
\]  

(4)

\[
\text{Phase angle (Aged)} = 40.12296 - 2.6113 \times A + 1.0360 \times B - 9.3012E - 003 \times A \times B + 0.56606 \times A^2 - 5.30175E - 003 \times B^2 - 0.022758 \times A^3
\]  

(5)
Tables 2–5 show the statistical analysis (ANOVA) for the developed G* and δ before and after ageing. The degree of correlation could be checked by the correlation coefficient (R²). From the tables, all responses had R² values of more than 0.97, which indicated the correlation error for these models was less than or equal to 3%. However, after removing insignificant terms in the models, R² was slightly reduced as Adj. R². Furthermore, the Adj. R² values for all models were consistent with the predicted R² values, with a difference of less than 0.2 [20]. The high R² values indicated that the RSM technique proposed was suitable for modelling the G* and δ of bio-binders with few samples and low errors. The P-value for complex modulus of all models, and their independent variables were checked to ensure that P was less than 0.05 to be statistically significant. The P-values for all models and most of their independent variables were less than 0.05, indicating that the cubic models were selected, and their terms were significant and within the confidence interval of 95%. For all models, F-value indicated that the models were significant.

To prove the developed models, some values of unaged bio-asphalt binders from Table 1 were selected to be compared with the predicted values calculated by equations 2-5 as shown in Table 6.

### Table 2. ANOVA results for complex modulus (unaged)

| Source | Sum of squares | df | Mean square | F Value | p-value prob > F |
|--------|----------------|----|-------------|---------|-----------------|
| Model  | 89.77          | 8  | 11.22       | 1691.30 | < 0.0001        | Significant |
| A-CPO  | 4.51           | 1  | 4.51        | 680.48  | < 0.0001        | R² = 0.9986 |
| B-Temperature | 26.32 | 1  | 26.32       | 3966.93 | < 0.0001       | Adj R² = 0.9980 |
| A²     | 1.39           | 1  | 1.39        | 208.87  | < 0.0001        | Pred. R² = 0.9946 |
| B²     | 0.059          | 1  | 0.059       | 8.96    | 0.0075          |
| A²B    | 0.011          | 1  | 0.011       | 1.66    | 0.2134          |
| AB     | 0.12           | 1  | 0.12        | 17.84   | 0.0005          |
| A³     | 1.01           | 1  | 1.01        | 151.66  | < 0.0001        |

### Table 3. ANOVA results for complex modulus (Aged)

| Source | Sum of squares | df | Mean square | F Value | p-value prob > F |
|--------|----------------|----|-------------|---------|-----------------|
| Model  | 99.90          | 8  | 12.49       | 2380.75 | < 0.0001        | Significant |
| A-CPO  | 6.39           | 1  | 6.39        | 1218.21 | < 0.0001        | R² = 0.9990 |
| B-Temperature | 23.78 | 1  | 23.78       | 4533.73 | < 0.0001       | Adj R² = 0.9986 |
| AB     | 0.14           | 1  | 0.14        | 26.77   | < 0.0001        | Pred. R² = 0.9972 |
| A²     | 0.50           | 1  | 0.50        | 95.29   | < 0.0001        |
| B²     | 0.034          | 1  | 0.034       | 6.45    | 0.0200          |
| A²B    | 0.072          | 1  | 0.072       | 13.66   | 0.0015          |
| AB²    | 0.080          | 1  | 0.080       | 15.22   | 0.0010          |
| A³     | 1.05           | 1  | 1.05        | 199.98  | < 0.0001        |

### Table 4. ANOVA results for phase angle (Unaged)

| Source | Sum of squares | df | Mean square | F Value | p-value prob > F |
|--------|----------------|----|-------------|---------|-----------------|
| Model  | 507.00         | 6  | 84.50       | 246.10  | < 0.0001        | Significant |
| A-CPO  | 82.02          | 1  | 82.02       | 238.86  | < 0.0001        | R² = 0.9860 |
| B-Temperature | 295.39 | 1  | 295.39      | 860.30  | < 0.0001       | Adj R² = 0.9820 |
| AB     | 22.03          | 1  | 22.03       | 64.16   | < 0.0001        | Pred. R² = 0.9700 |
| A²     | 2.19           | 1  | 2.19        | 6.38    | 0.00197        |
| B²     | 14.81          | 1  | 14.81       | 43.12   | < 0.0001        |
| A³     | 36.22          | 1  | 36.22       | 105.49  | < 0.0001        |
Table 5. ANOVA results for phase angle (Aged)

| Source      | Sum of squares | df | Mean square | F Value | p-value prob > F |
|-------------|----------------|----|-------------|---------|-----------------|
| Model       | 770.93         | 6  | 128.49      | 138.03  | < 0.0001        | Significant |
| A-CPO       | 155.27         | 1  | 155.27      | 166.80  | < 0.0001        |
| B-Temperature | 497.58      | 1  | 497.58      | 534.52  | < 0.0001        | R² = 0.9753 |
| AB          | 10.90          | 1  | 10.90       | 11.71   | 0.0026          | Adj R² = 0.9682 |
| A²          | 51.03          | 1  | 51.03       | 54.82   | < 0.0001        | Pred. R² = 0.9490 |
| B²          | 12.24          | 1  | 12.24       | 13.15   | 0.0016          |
| A³          | 101.97         | 1  | 101.97      | 109.54  | < 0.0001        |

Table 6. Validation of prediction models

| CPO (%) | Temp. | G*, observed | G*, Predicted | δ, observed | δ, predicted |
|---------|-------|--------------|---------------|-------------|--------------|
| 5       | 40    | 0.51         | 0.509         | 85.41       | 84.73        |
| 10      | 52    | 1.51         | 1.523         | 85.39       | 85.32        |

3.2. Response surface contour plots

Figure 1 shows the prediction versus actual values of G* and δ of unaged bio-binders. The plot aids in understanding the developed model behaviour. All points for the dependent variables (responses) are spread near the equality line, indicating the experimental results are consistent with the predicted results which reflect the precision of the models.

**Figure 1.** Predicted versus actual plots of unaged bio-asphalt (a) Complex modulus (unaged) (b) Phase angle

**Figure 2.** 2D contour plot of unaged bio-asphalt binders (a) G* (unaged) (b) δ
The 2D and 3D surface plots of the G* and δ models at different COP contents and various temperatures are presented in Figures 2 and 3, respectively. The curvature contour lines in shape imply the good correlation among the independent variables. The red regions in 2D or 3D indicate the high values of G* and δ. Therefore, the bio-asphalt binder maintains similar or higher values of G* at COP contents ranging from 0% to 5% compared with those of the base asphalt binder with a lower phase angle and same COP content. This result could be attributed to the compatibility of COP with the base asphalt binder at low content and the polymerization of COP at bitumen mixing temperature [21]. Thus, 5% COP could be identified as the optimum bio-oil percentage for asphalt binder modification. Generally, for COP contents of more than 5%, the complex modulus of all bio-asphalt binders decreases, whereas the phase angle increases with the test temperature and COP content. This result could be attributed to the incompatibility of the high amount of COP with the base bitumen. This finding is consistent with the results in [22], which showed similar G* and δ behaviours of bio-asphalt binder. The behaviour of aged bio-asphalt binders in the present study is similar with that of unaged binders in terms of the increase and decrease of the G* and δ with various COP contents.

3.3. Multi-objective optimization of response
Multiobjective optimisation was applied to achieve the optimum COP content, resulting in high complex modulus values. The aim for optimisation was set as follows: COP in the range of 0%–15%, temperature test in the range of 40 °C–76 °C, complex modulus of more than 1 kPa under the unaged condition and phase angle in the range of 73.13 to 88.9 based on the values predicted by the model. The optimum values were selected based on the highest COP content at a desirability of 1.00, which was 5% COP at 64 °C, as discussed in the experimental part of this study.

4. Conclusions
In this research, the RSM method was utilised to evaluate the influence of COP on the rheological characteristics of the bitumen with 60/70 penetration grade under unaged and RTFO-aged conditions. According to the results and analyses, the following conclusions can be highlighted:
- Statistical analyses showed that the predicted values acquired from the developed models are consistent with the actual results from the experimental work.
- The coefficient of correlation (R²) showed values of more than 0.97 for all developed models, indicating a high degree of fitting among the developed cubic models and the actual experimental data.
- A high G* of bio-asphalt binders was obtained with 5% COP at 64 °C, which is near the G* of the base bitumen. However, for COP of more than 5%, the G* decreased with the increase of COP concentration.
The optimisation result indicated that the developed models could predict the rheological properties of CPO-modified asphalt binders with acceptable accuracy.

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