Optimisation of Polymer Addition Using the Plackett-Burman Experiment Plan

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Abstract. At the present time, the utilization of waste polymer materials belongs to one of the most important challenges where global economies have to tackle. This article concerned the modification of petroleum road bitumen with waste polymer. The bitumen modification process with the use of polymeric materials was carried out considering a number of other quantitative factors, such as: mixing time, mixing speed, bitumen temperature and qualitative factors such as: waste polymer content, type of grain size, type of neat bitumen and type of waste polymer. Two kinds of waste polymers (PET, PP) were used in the research, which served as a modifier. Two petroleum bitumens were used: 20/30 (hard) and 70/100 (soft).

Based on the divalent Plackett-Burman experiment plan, the number of variables and the number of combinations of mixtures were determined, which were required to determine the final response surface model. The following features were tested as the output variables: penetration, softening point, Fraass breaking point, dynamic viscosity 60oC, 90oC, 135oC, deformation energy and maximum elongation. The use of the experimental design methodology allowed to identify the factors that had the greatest impact on the bitumen modification process. The assessment of the significance of the parameters also allowed to identify a significant model allowing to find the optimal bitumen and waste polymer composition. Based on the test results, it was shown that the consistency of the modified Plackett-Burman experiment plan, the number of variables and the number of combinations of mixtures were determined, which were required to determine the final response surface model. The following features were tested as the output variables: penetration, softening point, Fraass breaking point, dynamic viscosity 60oC, 90oC, 135oC, deformation energy and maximum elongation. The use of the experimental design methodology allowed to identify the factors that had the greatest impact on the bitumen modification process. The assessment of the significance of the parameters also allowed to identify a significant model allowing to find the optimal bitumen and waste polymer composition. Based on the test results, it was shown that the consistency of the modified bitumen was influenced by the type of bitumen, its amount, mixing speed and mixing time. With regard to the softening point, the type of polymer was also an important factor. Ultimately, the optimization process allowed for the determination of such a combination of both qualitative and quantitative input factors, which resulted in bitumen showing higher utility than input 20/30 and 70/100 bitumens. Moreover, it was found that the increase in mixing time did not result in an excessive increase in bitumen stiffness caused by the mixing process. Thus, the low-temperature properties left unchanged significantly.

1. Introduction

Road bitumen, which is an end product of distillation, has been used in road construction for many years [1]. The bitumen’s composition and temperature strongly affect its properties and microstructure. Due to the above, its use as a road binder is a very good solution for producing road composites [2]. The binder’s evaluation with the use of rheological properties is the correct tool describing the bitumen’s behaviour in the pavement [3]. There are many studies referred to by the Strategic Highway Research Program (SHRP), the main objective of which was to obtain information on the usefulness of a broad range of bitumen available in the market in the most effective manner.
This allows for controlling the rheology of ordinary and modified bitumen at high bitumen pumping temperatures, optimal binder-aggregate mixing as well as incorporation and compaction of bituminous composites [4]. Ordinary road bitumen do not always allow for obtaining a high-durability road pavement layer. Increasing traffic and heavy vehicle participation makes researchers shift their attention to bitumen with improved properties. A commonly used technology is to utilise the SBS copolymer modified bitumen with an increased range of viscoelasticity, which ensures the bituminous composite’s increased resistance against low-temperature cracking on one hand and reduces the risk of permanent deformations at high temperatures in the summer on the other hand [5]. Another solution that modifies base bitumen properties, especially in terms of the bituminous mixes’ increased resistance against permanent deformation as well as increased workability during production is the use of synthetic waxes or their presence during the foaming process [6]–[8]. Nevertheless, a small quantity of waxes or the SBS copolymer added to the base bitumen substantially increases the costs of investment execution. Due to the above, it is justified to seek other solutions that will allow for cost minimisation with simultaneous maintenance of the bituminous composite’s minimum performance requirements. One of such solutions is to use alternative polymer products (thermoplastic plastomers) [9]. Nevertheless, it is a complex task to select the type of polymer substitute material, bitumen and important factors controlling the mixing process. A correct modification process requires the use of a suitable sampling method. This paper presents the results of preliminary testing with the use of a saturated elimination experiment plan. It allowed for the formulation of the problem and establishment of a stable regressive model dependent on a series of factors. Furthermore, the applied Plackett-Burman experiment plan allowed for the evaluation of the significance of the impact of the given input factor, thereby enabling a preliminary optimisation of the bitumen modification process with the use of an alternative polymer material.

2. Materials
2.1. Bitumen
The preliminary stage of testing featured activities aimed at a complex evaluation and selection of bitumen, the properties of which would be modified with the selected polymers. According to the Plackett-Burman experiment plan methodology, the testing required the adoption of two different bitumen. Their selection was dictated by the need to represent the bitumen’s different rheological states. Due to the above, the testing featured a “gel” type bitumen with the penetration range of 20/30 and a “sol-gel” type bitumen with the penetration range of 70/100. The base bitumen underwent a series of basic and rheological testing. The same tests were also conducted for all bitumen mix compositions with alternative polymers. Table 1 presents the test results including references to the testing methods.

| Feature                                    | Neat bitumen | Referenced bitumen [10] | Standard               |
|--------------------------------------------|--------------|-------------------------|------------------------|
| Penetration at 25°C, 0.1 mm                | 27.3         | 82.9                    | 37                     |
|                                            | 25           |                         | PN-EN 1426             |
| Softening point T_{R&B}, °C                | 62.6         | 45.5                    | 55                     |
|                                            | 84           |                         | PN-EN 1427             |
| Fraass breaking point, °C                  | -10.4        | -17.3                   | -13                    |
|                                            | -7           |                         | PN-EN 12593            |
| Elongation, cm (declared)                  | 143          | 316                     | -                      |
|                                            | -            |                         | PN EN 14023            |
| Cohesive energy, J/cm²                     | -            | 0.066                   | -                      |
|                                            | -            |                         | PN EN 14023            |
| Viscosity at 60°C, Pas                     | 3624         | 112                     | 2250                   |
|                                            |              |                         | 4850                   |
| Viscosity at 90°C, Pas                     | 96.4         | 7.6                     | 84.2                   |
|                                            |              |                         | 114                    |
| Viscosity at 135°C, Pas                    | 2.4          | 0.5                     | 1.9                    |
|                                            |              |                         | 0.7                    |
| Viscosity at 150°C, Pas                    | 2.4          | 0.2                     | 0.9                    |
|                                            |              |                         | 0.4                    |

Table 1 also includes the test results for bitumen within the tested bitumen consistency range. Bitumen 35/50 and its mix with a 2% content of synthetic wax 35/50+2%SW were chosen for this
purpose. The main objective of this activity was to refer the results of the tested modified bitumen to other available road bitumen, including those used in the WMA technology [10], [11].

2.2. Plastomers
The modification of the road bitumen properties was achieved by adding two plastics. Two types of plastomers were chosen: PET (polyethylene terephthalate) and PP (polypropylene). The used plastics are classified as thermoplastic materials with a crystalline structure, wherein PET is characterised by a high pour point when compared to PP, thereby justifying the selection of these materials for testing.

3. Plackett-Burman experiment plan
In experimentation, the Plackett-Burman design is classified as an orthogonal saturated experiment plan. Its main task is the quick and effective evaluation of the importance of the impact of input variables. On this basis, it is possible to eliminate variables that are insignificant in terms of their impact on the technological process, but also to quickly optimise the input factors. It thereby allows for seeking the best solutions for the road bitumen mix and thermoplastic polymer materials. The applied experiment plan is characterised by the fact that the number of experiments was equal to the number of variables increased by 1. The orthogonal Plackett-Burman design’s statistical inference was conducted based on a linear regression equation (1):

\[ y = b_0 x_0 + b_1 x_1 + \cdots + b_k x_k \]  

where: \( b_i \) – experimental factor, \( x_i \) – (independent) input variable, \( y \) – (dependent) input variable.

The next stage was to determine the significance of factors \( b_j \) (for \( j>0 \)). In this design, it is assumed that the interaction effects are insignificant. Eight different combinations were used in the paper. The factor variance was estimated based on multiple experiments in the form of samples and information in which dummy variables were the medium [12]. Due to the occurrence of qualitative variables, it was required to assign binary values to them. For this reason, the final model was determined by using the Generalised Linear Model [13].

The experiment plan assumed controlling 7 input variables on two levels during the preparation of 8 different mix combinations. The input variables used in the experiment featured a quantitative and qualitative nature. The breakdown of the variables and their respective levels are presented in Table 2.

| Variable                  | Type       | Unit          | Code | Low level/ Level one | High level/ Level two |
|---------------------------|------------|---------------|------|-----------------------|-----------------------|
| Mixing speed              | quantity   | rpm/min       | A    | 120                   | 9500                  |
| Mixing temperature        | quantity   | °C            | B    | 160                   | 180                   |
| Mixing time               | quantity   | min.          | C    | 30                    | 180                   |
| Waste polymer content     | quantity   | %             | D    | 2                     | 5                     |
| Bitumen type              | quality    | -             | E    | 20/30                 | 70/100                |
| Waste polymer type        | quality    | -             | F    | PP                    | PET                   |
| Waste polymer granulation | quality    | -             | G    | ≤5.6mm                | >5.6mm                |

The establishment of the ranges of values assigned to particular variables was based on literature analyses [14], [15]. In the case of qualitative variables, the scope was established to reflect the high-speed and low-speed mixing process. The plastomer content was limited to 5% to prevent the occurrence of high dynamic viscosity >3Pas at 135°C [9]. In addition, the impact of the granularity of the plastomer responsible for the composition’s homogenisation was taken into account.
4. Analysis of the modification process

The modification process was conducted in accordance with the test program presented in Chapter 3. It is necessary to remember that subsequent combinations were prepared and tested with maintenance of complete randomisation. This allowed for the avoidance of an additional systematic error. The test results along with the combinations assigned to the given experiment plan are presented in Table 3.

Table 3. Results of property designation for bitumen modified based on the Plackett-Burman designs

| Case | A | B | C | D | E | F | G | T<sub>R&B</sub> | Penetration | Fraass | Cohesion | Elongation | Dynamic viscosity |
|------|---|---|---|---|---|---|---|------------|------------|--------|-----------|-------------|-----------------|
|      | rpm/min<sup>1</sup> | °C | min. | % | - | - | - | °C | 0.1mm | °C | E J/cm<sup>2</sup> | L mm | η<sub>135</sub> Pas |
| 1    | 120 | 160 | 30 | 5 | 70/100 | PP | <5.6 | 53.7±3.7 | 72.6±0.8 | -9.9±2.2 | 0.134 | 283±161 | 0.53±0.01 |
| 2    | 120 | 160 | 180 | 5 | 20/30 | PET | >5.6 | 78.8±7.5 | 28.2±0.8 | -10.7±0.4 | 0.000 | 84±14 | 2.2±0.02 |
| 3    | 120 | 180 | 30 | 2 | 70/100 | PET | >5.6 | 54.4±4.5 | 86.7±1.8 | -12.7±2.3 | 0.098 | 290±146 | 0.45±0.01 |
| 4    | 120 | 180 | 180 | 2 | 20/30 | PP | <5.6 | 59.1±5 | 21.3±1.7 | -9.7±1.3 | 0.000 | 97±16 | 3.7±0.1 |
| 5    | 9500 | 160 | 30 | 2 | 20/30 | PP | >5.6 | 56±4.5 | 19±3.5 | -10±1 | 0.000 | 67±16 | 3.32±0.01 |
| 6    | 9500 | 160 | 180 | 2 | 70/100 | PET | <5.6 | 55.2±4.3 | 76.6±2.5 | -11.9±1.7 | 0.117 | 481±270 | 0.53±0.01 |
| 7    | 9500 | 180 | 30 | 5 | 20/30 | PET | <5.6 | 78.6±7.6 | 28±1 | -8.4±1 | 0.000 | 146±52 | 2.3±0.16 |
| 8    | 9500 | 180 | 180 | 5 | 70/100 | PP | >5.6 | 117.8±20 | 21.4±2.5 | -10.7±0.9 | 0.000 | 70±2 | 2.89±0.07 |

During a preliminary analysis of the partial test results, it is necessary to note that the penetration value decreases along with an increase in the mixing time and mixing speed. The bitumen and polymer mixing temperature is also of importance. An inverse correlation was observed in the case of feature T<sub>R&B</sub>, which is directly correlated with the bituminous mixes’ potential permanent deformation resistance. It must be noted that cohesion energy other than zero was obtained in case 6. This was possible because in several cases it was possible to achieve a bitumen elongation of 1333% (400mm) at 10°C when compared to the starting value. It is important to note that the Fraass breaking point was maintained at the same level within the results’ confidence range. The measurement results for all mixes in accordance with the Plackett-Burman design along with the reference results (20/30, 70/100, 35/50 and 35/50+2.5%SW) are presented in Figure 1.

The results presented in Figure 1 indicate in a meaningful way that the bitumen modification with the selected plastomers affects the bitumen’s properties. It was significant that some bitumen modification combinations allowed for obtaining a solution similar to the results obtained for bitumen 35/50+2.5% of synthetic wax (SW). This suggests that it is possible to obtain bitumen with properties similar to synthetic wax modified bitumen in low and medium operating temperatures, except that the Fraass breaking point was lower in some by cases by as much as 5°C. It is of great significance in terms of the low-temperature cracking of bituminous composites made from this type of bitumen. It must be noted that the modification of bitumen using plastomer resulted in the output bitumen having a consistency in between that of bitumen 20/30 and bitumen 70/100, and similar to bitumen 35/50. It was significant that the softening point in bitumen modified using thermoplastic plastomer increased regardless of the case and was at least at the level of results of T<sub>R&B</sub> for bitumen 35/50 and was able to achieve values >100°C (case 8). When referring the dynamic viscosity η<sub>135</sub> designated at 135°C to the results, its values allowed for the aggregate’s adequate coating in nearly all cases. A summary of the observations lead to the conclusion that the input bitumen’s consistency affected the aforementioned features. A variance analysis (ANOVA) was used for the quantitative determination of the force and trend of impact of the given independent variable on the measured (dependent) variable. Figure 2 utilises Pareto charts to graphically present the impact of the factors on the resulting variable.
The results presented in Figure 2 confirm the previous hypothesis that the type of bitumen has the greatest impact on the given feature. Its negative value means that the greater the base bitumen’s penetration, the greater the modified bitumen’s penetration. This fact results from the adoption of the bitumen type as the qualitative variable. In the case of feature $T_{R&B}$, the bitumen type turned out to be the least important, despite of its substantial impact. In terms of the other variables, the granulation and polymer also played a very important role in the creation of quality of the final plastomer modified bitumen. In the case of the mixing temperature, its range from 160°C to 180°C was the least important on the output bitumen’s properties. In case of features $T_{R&B}$ and Penetration, the mixing time and rotation speed were also of significance in terms of the creation of structure of plastomer modified bitumen. An interesting observation is that the explanation of the Fraass breaking point’s variability does not feature two variables, i.e. bitumen type and polymer granulation. The exclusion of the bitumen temperature and mixing time from the factors substantially affecting the breaking point leads to the assumption that the bitumen’s ageing process was of secondary importance in this experiment. In relation to the dynamic viscosity, aside from the bitumen and plastomer type as well as the mixing time, the bitumen’s temperature was of great importance in creating its value. On the other hand, the polymer content was of secondary importance when compared to viscosity at high temperatures.

**Figure 1.** Breakdown of results for modified and reference bitumen mixes.
Figure 2. Quantitative evaluation of the impact of input variables on the output variable: a) Penetration; b) $T_{RRB}$; c) Fraass breaking point, d) elongation (declared), e) dynamic viscosity ($\eta_{135}$).

The quantitative evaluation of the main effects using the ANOVA linear model was aimed at testing whether there is basis for seeking the test subject’s important function that would describe all dependent variables (measured variables). In effect, it must be noted that at least one of the input variables had a substantial impact on the variability of the input variables. For this reason, the identification of the test subject’s function parameters was commenced. The table below presents the results of matching the linear model in accordance with formula (1). Table 4 presents the results of matching the model with the experimental values.

In the case of variable E, the matching value for the modified factor $R^2$ was not determined. This resulted from the insufficient number of samples, in relation to other tests, which prevented the determination of a reliable repeatability error. Therefore, the model parameter identification describing the cohesion energy E was conducted based on the average values in nodes at different sample sizes. For other variables, the value of the significant factor $R^2$ was determined in an acceptable range of 0.66-0.98 at an acceptable mean square error MSE. The last stage of analysis, i.e. the modification process optimisation, was commenced based on important identified mathematical regression models. With an established set of criteria, its objective was to determine the most suitable configuration of input variables for obtaining a satisfactory output product in the form of plastomer modified bitumen [18]. The criteria were defined for this purpose and their results are presented in Table 5.
Table 4. Parameters of the linear regression model

| Variable                        | Penetration\[^a\] | TR&B\[^a\] | T\[^o\] | E\[^a\] | L\[^a\] | η\[^135\] |
|---------------------------------|-------------------|-----------|--------|--------|--------|-----------|
|                                 | 0.1 mm            | °C        | °C     | J/cm\(^2\) | mm    | Pas       |
| Intercept                       | 44.3042           | 67.8234   | -10.7833 | 0.043562 | 189.8733 | 1.9926    |
| (1) Mixing speed                | -8.3708           | 13.1487   | -0.0083 | -0.014312 | 1.1808  | 0.2657    |
| (2) Mixing temperature          | -5.1292           | 12.7188   | 0.1333  | -0.019063 | -39.0683 | 0.3476    |
| (3) Mixing time                 | -7.8792           | 13.1694   | -0.7417 | -0.014312 | -6.7808  | 0.3430    |
| (4) Waste polymer content       | -7.0542           | 12.8279   | 0.6583  | -0.010187 | -43.9358 | -0.0140   |
| (5) Bitumen type                | -19.6958          | -4.1272   | 1.7167  | -0.043562 | -91.3017 | 0.8959    |
| (6) Waste polymer type          | 10.6958           | -13.4365  | -0.5917 | 0.010188  | 60.5142  | -0.6251   |
| (7) Waste polymer granulation   | -3.7078           | 12.7481   | -1.1333 | -0.019063 | -61.9567 | 0.2211    |
| R\(^2\) [16]                    | 0.98              | 0.98      | 0.66    | -       | 0.68    | 0.99      |
| MSE [17]                        | 2.9               | 4.4       | 2.1     | -       | 52      | 0.053     |

\[^a\] - values marked in “Italic” designated as significant

Table 5. Limit ranges of utility profiles (criteria)

| Level                | Penetration\[^a\] | TR&B\[^a\] | T\[^o\] | E\[^a\] | L\[^a\] | η\[^135\] |
|----------------------|-------------------|-----------|--------|--------|--------|-----------|
|                      | 0.1 mm            | °C        | °C     | J/cm\(^2\) | mm    | Pas       |
| Low (value 0)        | <45               | <65       | >-5    | 0      | <200   | >3        |
| High (value 1)       | >80               | >80       | min    | max    | >400   | <1        |

Figure 3. Results of the optimisation of bitumen modification using plastomer
The main objective was to obtain bitumen with high $T_{R&B}$ similar to bitumen $PmB_{45/80-65}$, which does not demonstrate problems with aggregate coating at high temperatures ($\eta_{135}$). Furthermore, plastomer modified bitumen should demonstrate low penetration, low breaking point ($>-5^\circ\mathrm{C}$) higher than the result obtained for bitumen $35/50+2.5\%\mathrm{SW}$. The search assumed a solution with the maximum possible strain energy and a bitumen that would achieve elongation of up to 1333% at 10°C. The “min” and “max” values mean that the criterion was taken from the range of results from the given experiment for the given variable. As result, an optimisation process was conducted, which resulted in obtaining the utility function (objective function), which in essence constituted a geometrical mean [12]. The optimisation result is presented in Figure 3.

Based on the conducted optimisation in experiment plan’s mesh nodes, it was discovered that obtaining a good (0.77) utility function result above the average (>0.5) required the use of the following settings:

- Rotation speed: 9,500 rpm/min$^1$;
- Bitumen temperature >175°C
- Mixing time: 180 minutes;
- Waste polymer quantity: 3.5%;
- Road bitumen: soft (70/100);
- Plastomer type: including features of PET type polymer or similar;
- Granulation: fine (<5.6mm)

As result, the bitumen produced in accordance with the aforementioned control variables demonstrated properties which were as follows when compared to comparative bitumen (Fig. 4)

![Figure 4](image-url)
The horizontal lines in Figure 4 represent the probable optimal solution for the given feature at the criteria assumed in Table 5, estimated with a probability of 95%. It must be noted that the modified bitumen produced in optimal conditions (determined in the experiment) confirms the hypothesis that it is possible to obtain bitumen with more favourable properties than those of the tested comparative bitumen (Fig. 4). Plastomer modified bitumen demonstrates high penetration at maintained high softening point when compared to road bitumen. Its softening point was slightly lower than that of the synthetic wax modified bitumen (35/50 + 2.5%SW). Nevertheless, plastomer modified bitumen demonstrated a substantially lower breaking point. This result is key from the point of view of maintaining the bituminous mixes’ high durability at low temperatures. Furthermore, this bitumen can potentially achieve an elongation of 1333% during a ductility test. It was therefore possible to estimate its small cohesion energy. This was another property that substantially distinguished it from road bitumen.

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5. Conclusions
The following conclusions were formulated based on the conducted tests and analyses:

- The Plackett-Burman design turned out to be a perfect tool that enabled quick evaluation of the entire process of bitumen modification using plastomer. It also allowed for the formulation of an optimal solution.
- The use of plastomers guarantees obtaining modified bitumen with properties more favourable than those of road bitumen. The polymer modified bitumen achieved a higher softening point while maintaining a low Fraass breaking point. Furthermore, the presence of polymer allowed for the measurement of the non-zero cohesion energy. This fact demonstrated the great potential of polymer modified bitumen in the production of durable bituminous composites.
- Bitumen consistency, polymer granulation and type turned out to be the most important parameters during bitumen modification using plastomer. On the other hand, the rotation speed and mixing time turned out to be of secondary significance, though still important.
- The testing demonstrated the low impact of temperature during mixing on the increase in the Fraass breaking point. Thereby, the technological ageing process turned out to be a factor of lesser importance.
- In most cases and in the optimal solution, the presence of polymer achieved $\eta_{1,35} < 3 \text{Pas}$, thereby having a positive impact on the bitumen’s homogenisation of thermoplastic plastomer with the aggregate.

References
[1] P. Partal, F. Martinez-Boza, B. Conde, i C. Gallegos, „Rheological characterisation of synthetic binders and unmodified bitumens”, Fuel, t. 78, nr 1, s. 1–10, sty. 1999, doi: 10.1016/S0016-2361(98)00121-5.
[2] Y. R. Kim, Red., Modeling of asphalt concrete. Reston, VA : New York: ASCE Press ; McGraw-Hill, 2009.
[3] I. A. Hussein, M. H. Iqbal, i H. I. Al-Abdul-Wahhab, „Influence of Mw of LDPE and vinyl acetate content of EVA on the rheology of polymer modified asphalt”, Rheol Acta, t. 45, nr 1, s. 92–104, wrz. 2005, doi: 10.1007/s00397-005-0455-2.
[4] X. Lu, U. Isacsson, i J. Ekblad, „Rheological properties of SEBS, EVA and EBA polymer modified bitumens”, Mat. Struct., t. 32, nr 2, s. 131–139, mar. 1999, doi: 10.1007/BF02479440.
[5] R. Blanco, R. Rodríguez, M. García-Garduño, i V. M. Castaño, „Morphology and tensile properties of styrene–butadiene copolymer reinforced asphalt”, J. Appl. Polym. Sci., t. 56, nr 1, s. 57–64, kwi. 1995, doi: 10.1002/app.1995.070560108.
[6] M. F. C. Van de Ven, K. J. Jenkins, J. L. M. Voskuilen, i R. Van den Beemt, „Development of (half-) warm foamed bitumen mixes: state of the art”, *International Journal of Pavement Engineering*, t. 8, nr 2, Art. nr 2, cze. 2007, doi: 10.1080/10298430601149635.

[7] M. Iwański, G. Mazurek, P. Buczyński, i J. Zapala-Sławeta, „Multidimensional analysis of foaming process impact on 50/70 bitumen ageing”, *Construction and Building Materials*, s. 121231, paź. 2020, doi: 10.1016/j.conbuildmat.2020.121231.

[8] M. M. Iwanski, A. Chomicz-Kowalska, i K. Maciejewski, „Impact of Additives on the Foamability of a Road Paving Bitumen”, *IOP Conf. Ser.: Mater. Sci. Eng.*, t. 603, s. 042040, wrz. 2019, doi: 10.1088/1757-899X/603/4/042040.

[9] M. García-Morales, P. Partal, F. J. Navarro, F. Martínez-Boza, M. R. Mackley, i C. Gallegos, „The rheology of recycled EVA/LDPE modified bitumen”, *Rheologica Acta*, t. 43, nr 5, s. 482–490, lis. 2004, doi: 10.1007/s00397-004-0385-4.

[10] M. Iwański i G. Mazurek, „Optimization of the Synthetic Wax Content on Example of Bitumen 35/50”, *Procedia Engineering*, t. 57, s. 414–423, 2013, doi: 10.1016/j.proeng.2013.04.054.

[11] M. Cholewińska, M. Iwański, i G. Mazurek, „The Impact of Ageing on the Bitumen Stiffness Modulus Using the Cam Model”, *The Baltic Journal of Road and Bridge Engineering*, t. 13, nr 1, Art. nr 1, mar. 2018, doi: 10.3846/bjrbe.2018.386.

[12] Ž. R. Lazić, *Design of experiments in chemical engineering: a practical guide*. Weinheim ; [Germany]: Wiley-VCH, 2004.

[13] D. C. Montgomery, *Design and analysis of experiments*, Eighth edition. Hoboken, NJ: John Wiley & Sons, Inc, 2013.

[14] A. Modarres i H. Hamedi, „Effect of waste plastic bottles on the stiffness and fatigue properties of modified asphalt mixes”, *Materials & Design*, t. 61, s. 8–15, wrz. 2014, doi: 10.1016/j.matdes.2014.04.046.

[15] L. Brasileiro, F. Moreno-Navarro, R. Tauste-Martínez, J. Matos, i M. Rubio-Gámez, „Reclaimed Polymers as Asphalt Binder Modifiers for More Sustainable Roads: A Review”, *Sustainability*, t. 11, nr 3, s. 646, sty. 2019, doi: 10.3390/su11030646.

[16] R. F. Bonaquist, *Refining the simple performance tester for use in routine practice*. Washington, D.C: Transportation Research Board, 2008.

[17] G. Mazurek, „Analysis of selected properties of asphalt concrete with synthetic wax”, *Bulletin of the Polish Academy of Sciences Technical Sciences*, t. 66, nr 2, Art. nr 2, 2018, doi: 10.24425/122102.

[18] M. Iwański, G. Mazurek, i P. Buczyński, „Bitumen Foaming Optimisation Process on the Basis of Rheological Properties”, *Materials*, t. 11, nr 10, Art. nr 10, wrz. 2018, doi: 10.3390/ma11101854.