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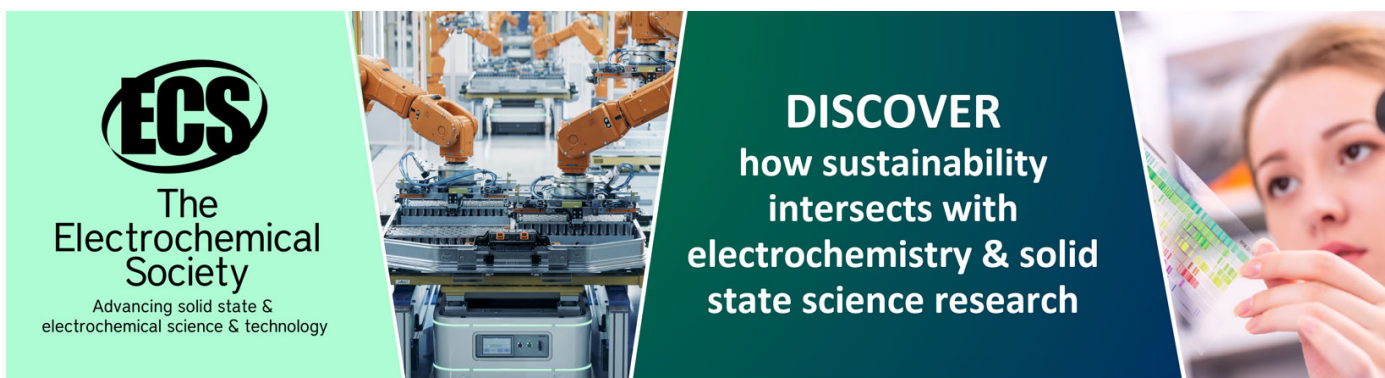
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Optimisation of the innovative hydraulic binder composition for its versatile use in recycled road base layer

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Abstract. The paper presents results obtained on the basis of tests carried out using the innovative hydraulic binder intended for a recycled base layer with foamed bitumen. The tested hydraulic binder consisted of three components: cement, hydrated lime and fines. The analysis includes: final time of mortar setting, volume change in mortar, compressive strength after 7 and 28 days of curing, bending strength after 7 and 28 days of curing. The analysis relied in identifying optimal compositions of innovative binder components in terms of its suitability for pavement recycled base layer. Optimisations considered obtaining a two variant such as: quick-setting and a normal-setting hydraulic binder. The analysis of test results indicate that there are solutions for binder compositions that meet the requirements for normal-setting of hydraulically bound base layer in recycled technology. In the scope of obtaining a quick-setting binder, at least 80% of cement binder in the innovative hydraulic binder composition is required.

1. Introduction

The pro-environmental policies introduced around the world contribute to the development and application of technologies ensuring the lowest attainable power consumption, limitation of pollution and natural environment degradation. There is a series of industry disciplines that generate substantial pollution resulting from overproducing by-products. Such processes include: road construction - dismantling of destroyed pavements, production of mineral and asphalt mixtures, production of mineral aggregates and cement. One of the solutions effective in terms of pollution contamination is the introduction of warm mineral asphalts (WMA)[1]. Another important source of pollution features mineral and asphalt mixture production plants releasing substantial quantities of dust into the atmosphere. One of the methods of limiting their emission is the use of bag filters [2]. Deep cold recycling using foamed asphalt provides potential ability of dust utilisation [3], [4]. A similar problem occurs in the case of utilisation of some dusts generated during the Portland cement production. The use in cement plants of dedusting devices that substantially limit dust emission is also related to the need of maintaining sufficient level of raw material and air flow in the cement production system. These are important problems, because cement production using the most popular dry method generates dust at every stage of the process. In terms of production operations featuring dust generation, dust can be divided into related with processes and transport. These include dust from the dedusting system (Cement Kiln Dust - CKD) and from by-passing (Cement By-pass Dust – CBPD) rotary kilns for firing Portland clinker. The composition of dust generated in the kiln dedusting system differs from the composition of



raw meal fed into the kiln, because it is generated along the entire length of the kiln's drum. The domestic and foreign guidelines do not refer to the use of additives for the mixed hydraulic binder generated along with, e.g. Portland cement. The use in research of an innovative binder for a substructure recycled using the deep "cold" recycling technology can constitute an alternative solution for a traditional binder, such as the Portland cement. This way, during the recycled substructure composition planning stage, it is possible to take into consideration the environmental conditions in which the substructure will operate [5]. One of the conditions set for new mixed binders is ensuring the required physical and mechanical properties of the recycled substructure. The market features a series of road binders that are produced in cement plants based on a recipe constituting a trade secret or subject of a patent restriction.

Literature analysis confirmed the validity of conducting further testing over compiling binders with the use of waste material dust. Many researchers are seeking answers in the scope of using additives (e.g. fly ash, cement ash, coal firing ash) in cement as binders in the composition of recycled substructures [6], [7]. However, these are singular component combinations. The Kielce University of Technology is conducting research on the attempt to introduce mixed binders in improved subsoil and substructures from hydraulically bound aggregates [8], [9]. The results obtained from the conducted research confirm the validity of continuing the testing of mixed binders for road applications, which can be used in the deep recycling technology with the use of foamed asphalt [10]. This technology features activities aimed at developing an innovative binder in the form of a hydraulic binder intended for mineral and binder mixtures that can be used in the deep cold recycling. This technology also covers the use of foamed asphalt. The necessity to use an innovative binder in the composition of a recycled substructure (hydraulic binder) will be high if the existing substructure features a very low-quality aggregate (high content of clay-silt particles, low physical parameters).

2. Materials and methods

2.1. Materials used for preparing the binder

The preparation of all compositions of a universal binder featured the use of three types of starting materials, i.e. CEM I cement (C), hydrated lime (HL) and cement bypass dust (CBPD). The chemical compositions of starting materials are presented in table 1.

Table 1. Comparison of the chemical composition of the C and CBPD components

Material	Composition, %											
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	Na ₂ O _e	Cl	SO ₃	LOI	
C	19.70	4.28	2.44	64.50	1.60	0.14	0.79	0.66	0.043	3.33		
CBPD	17.49	3.88	2.01	59.08	1.49	0.29	6.83			1.87	16.31	

In the case of cement bypass dust, there are two phases demonstrating binding properties: CaO and C₂S as well as sylvine (potassium chloride) and calcite. The components can derive from the feed after pre-calcination and could be moved from the kiln's interior by the air stream carrying the bypassed chlorine compounds. Additionally, the analysis of the granulation curve indicates that the HL and C granulations are similar and differ from CBPD. Nevertheless, the HL granulation is slightly more fine-grained than of the C component. The CBPD granulation is finer than of C and HL.

2.2. Slurry and mortar testing methods

The experiment featured 4 types of testing that, based on standard scopes, were used to optimise the universal binder's composition. Firstly, it was necessary to determine the water-binder ratio for obtaining slurry and mortar with a standard texture for particular binders. The slurry texture was determined by testing with the use of the Vicata instrument, whereas the mortar texture – using the concrete slump test and flow table test. Depending on the type of binder, the mortar preparation featured

the CEN standard sand compliant with the requirements of the PN-EN 196-1:2005 standard [11]. Based on the results of the mortar texture tests and determination of the correct quantity of water in slurry, it was ascertained that substituting cement with hydrated lime or cement bypass dust results in an increase in the binder's water demand. The presence of the cement bypass dust increased the water demand more than the addition of hydrated lime. The following standard parameters were designated during the determination of the water/binder ratio in relation to the standard mortar texture:

- start of setting acc. to PN-EN 196-3 [12],
- swelling acc. to PN-EN 196-3 [12];
- compressive strength after 7 and 28 days of curing acc. to PN-EN 196-1 [11],
- bending strength after 7 and 28 days of curing acc. to PN-EN 196-1 [11].

The standard requirement [12] for a cement binder specifies that the change in volume tested in Le Chaterlier's ring should not exceed 10 mm. This condition is met for C/HL/CBPD = 1.0/0/0 and the binder including C/HL/CBPD = 0.4/0.4/0.2. On the other hand, all other binder compositions demonstrate a swelling effect. The testing of the mortars' tensile strength at bending were conducted after 7 and 28 days of curing on beams with the dimensions 40 × 40 × 160 mm, according to the standard [11].

3. Binder composition optimisation based on the mixture plan

In principle, matching the response surfaces with the results for mixtures is done the same way as matching the surfaces to data, e.g. from the central composition plan. However, there is a problem consisting in that the data for mixtures are limited, i.e. the sum of all components must be constant and equal to 100%. The mixture of three components can simultaneously be determined by specifying a point in a system of triangular coordinates defined by three variables. All experiment plans based on the mixture plan require apex points, i.e. mixtures consisting of only one component. In practice, these systems can be impossible to obtain due to costs or other limitations resulting from technological aspects. This experiment used mixture plans with limitations, i.e. the basic mixture plan was modified so that the quantity of each of the components is equal to 20% to 60%. Ultimately, the testing program was subjugated to the limited mixture plan based on a simplex centroid plan [13], [14]. The experiment's plan encompassed the preparation of 7 various universal binder compositions represented by the cases provided in table 2.

Table 2. Quantity of components of a universal binder based on the limited mixture plan

Component combinations	Components		
	Hydrated Lime (HL)	Cement (C)	Cement By-pass Dust (CBPD)
7 C(2)	0.33	0.33	0.33
6 C(1)	0.40	0.40	0.20
5 C(1)	0.40	0.20	0.40
1 V	0.20	0.20	0.60
4 C(1)	0.20	0.40	0.40
3 V	0.20	0.60	0.20
2 V	0.60	0.20	0.20

The location of the determined 7 mixed binder compositions according to table 2, after mapping in the experiment's plan is presented in figure 1.

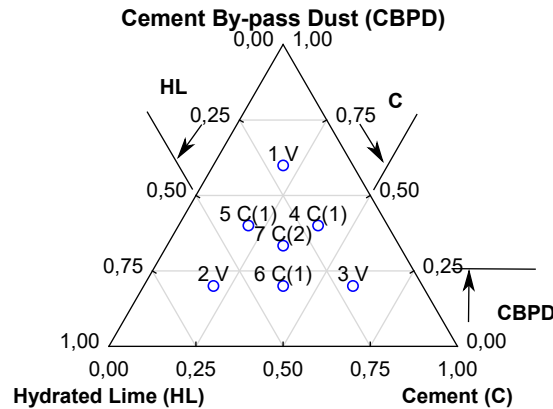


Figure 1. Limited mixture experiment plan

Aside from the locations of code marks of universal binder compositions, figure 1 also presents the method of determining the components' percentage value. According to figure 1, the given component quantity in the triangular plan is the length of the section constituting the bisection of the angle between the adjacent mixture plan triangle side. The direction designated by the arrow means the increase in the given component's percentage share in the binder. The use of the limited mixture plan is based on the estimation of pseudo-components, treating thereby the limited triangle as if it was a complete plan. The analysis of experiences from using mixture plans is in practice a multiple (multi-dimensional) regression with a fixed component reduced to zero. The evaluation of the mixture composition's impact on the properties of the innovative binder was determined based on the following algorithm:

- Analysis of adequacy of the type of approximated test object function.
- Estimation of the test object function coefficients.
- Evaluation of the significance of the approximated test object function coefficients.
- Analysis of the remnants.

A polynomial function was adopted as the approximating function. The polynomial degree depended on the significance of the contribution made by its form to explaining the test results' variability. The next stage of the analysis was the estimation of coefficients of the polynomial with the degree determined based on a variance analysis. The parameter approximation was based on the least squares method (LSM). The test object function's general model was as follows (1):

$$y = b_1x_1 + b_2x_2 + b_3x_3 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3 + b_{123}x_1x_2x_3 \quad (1)$$

where: b_{ijk} - the model's experimental parameters; x_i - i independent variable; y - dependent variable.

The stage featured an evaluation of the parameters' significance by using a student's test based on the intragroup variability dependent on the standard (sample) designation replication required by the conditions [11], [12]. The conducted preliminary analysis of the group indicated to a lack of bases for denying the hypothesis on the test results distribution compliance with the normal distribution. Finally, the regression model analysis featured an evaluation of the remnants' randomness aimed at indicating whether there is an unfavourable correlation between the average values and the corresponding standard deviations.

As already mentioned, the experiment plan based on the limited mixture plan consisted of 7 universal binder compositions constituting independent variables controlling the variability of six selected dependent features. The dependent features represent the standard tests of mortars prepared with the aforementioned binders (compositions) and include the following: a) start of setting, b) change in volume; c) compressive strength after 7 and 28 days of curing. The bending strength after 7 and 28 days of curing was a declared parameter. The optimal solution is a result of the optimisation process taking into consideration preconceived criteria. A change in a criterion substantially changes the result of estimating the desired results in terms of the material's properties. The aim of optimisation was to determine the usefulness of normally setting binders in the light of PN-EN 13282-2:2015-06 [15] fast-setting binders acc. to PN-EN 13282-1:2013-07 [16]. The optimisation criteria are presented in table 3.

Table 3. Standard criteria used for binders in the optimisation process

Normally setting [15]					
Standard designation	Compressive strength		Start of setting	Volume stability	
	[MPa]				[min]
	after 28 days			[mm]	
HRB-N1	≥ 2.5	≤ 22.5	≥ 150	≤ 30	
HRB-N2	≥ 12.5	≤ 32.5	≥ 150	≤ 30	
HRB-N3	≥ 22.5	≤ 42.5	≥ 150	≤ 30	
HRB-N4	≥ 32.5	≤ 52.5	≥ 150	≤ 30	
Fast-setting [16]					
	after 7 days		after 28 days		
HRB-E2	≥ 5.0	≥ 12.5	≤ 32.5	≥ 90	≤ 10
HRB-E3	≥ 10.0	≥ 22.5	≤ 42.5	≥ 90	≤ 10
HRB-E4	≥ 16.0	≥ 32.5	≤ 52.5	≥ 90	≤ 10
HRB-E4-RS	≥ 16.0	≥ 32.5	-	≤ 90	≤ 10

The optimisation process was conducted according to the usefulness function utilisation methodology proposed by Harrington [13]. Due to the presence of various input variables, it was necessary to subject their scope to standardisation in the $<0;1>$ interval. Partial usefulness profiles (functions) were used for this purpose. The usefulness profile function waveform is presented in figure 2.

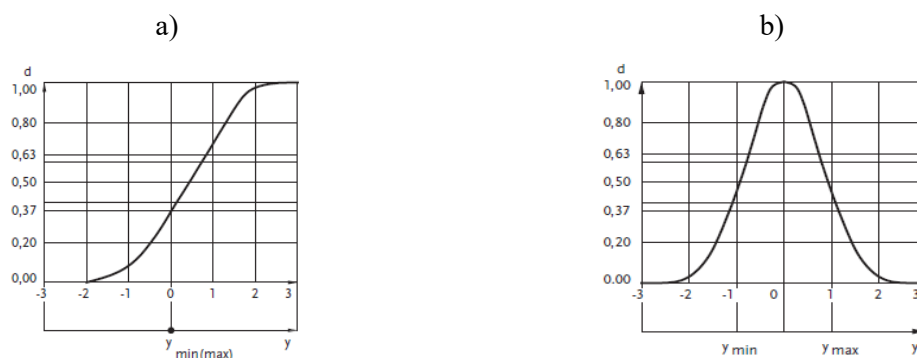


Figure 2. Usefulness profiles [14]: a) unilateral; b) bilateral

The evaluation of the mortars' compressive strength after 28 days featured the use of the symmetric profile (figure 2b), whereas the other cases utilised the unilateral profile (figure 2a). The generalised usefulness designated as D (also designated as $UIII$) is adopted as a weighted geometric average of particular d_u (2):

$$D = [\prod_{u=1}^n d_u]^{1/n} \quad (2)$$

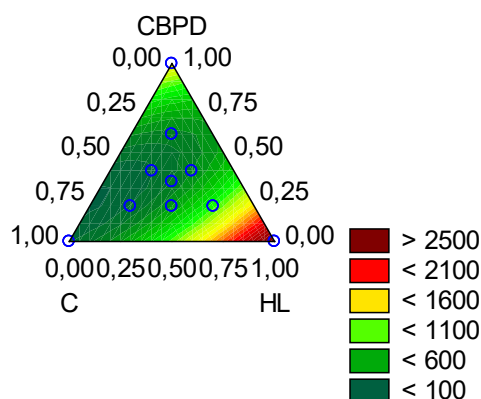
where: n- number of variables, d_u – partial usefulness functions.

The optimisation task was to search for a solution or area of solutions, in which the usefulness function has the minimum value of 0.37. The optimisation results obtained in the <0.37;0.63> interval are deemed as satisfactory solutions. On the other hand, results of >0.63 must be treated as a good result, where it is expected to obtain a material with above-average features.

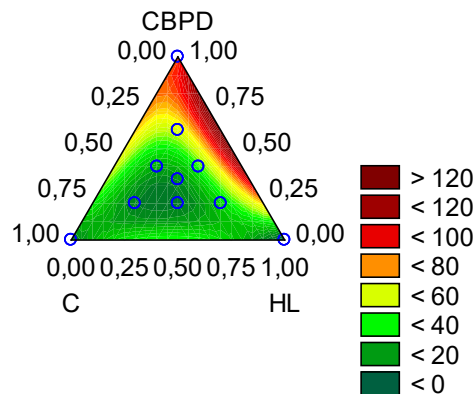
4. Test results

All universal binder compositions were made in quantities required by the standard procedures. The results were compiled in a graphical form along with the response surface model obtained for the given feature. The results are presented in a graphical form in figure 3.

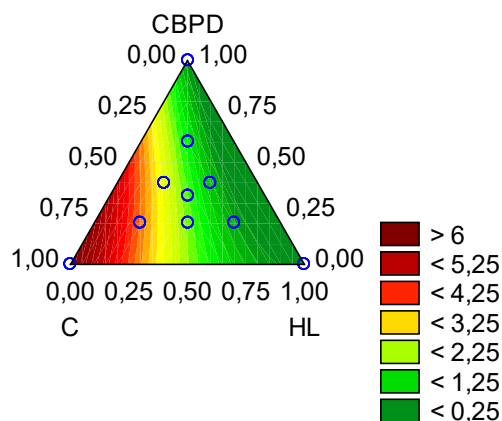
a)



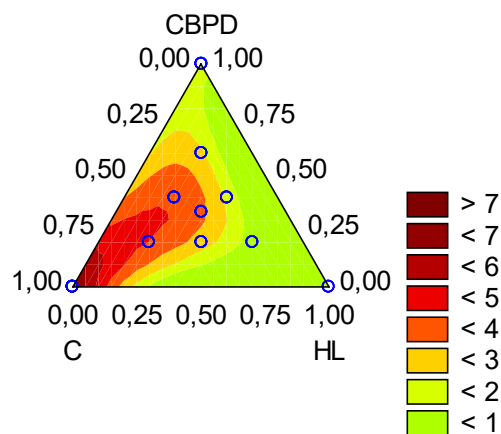
b)



c)



d)



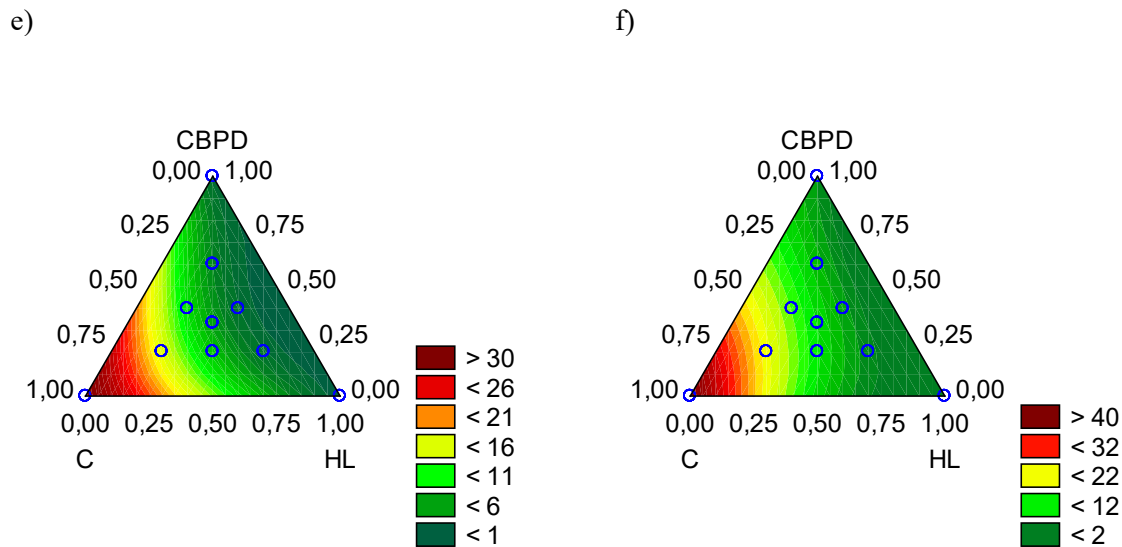


Figure 3. Results of the response surface based on the mixture plan: a) start of setting [s]; b) change in volume [mm]; c) bending strength after 7 days [MPa] d) bending strength after 28 days [MPa]; e) compressive strength after 7 days [MPa]; f) compressive strength after 28 days [MPa]

When analysing the results presented in figure 3, it is worth noticing that the highest compressive strength is demonstrated by samples including, as expected, large amounts of cement $C > 50\%$. On the other hand, the lowest strength would be demonstrated by compositions including 100% of lime or consisting solely of HL and CBPD. In the case of the bending strength, its increase after 28 days is unexpectedly intensified by the presence of a certain quantity of CBPD $> 40\%$ and $C > 50\%$. The presence of CBPD $< 20\%$ and cement $> 80\%$ resulted in obtaining a two-component binder that would demonstrate the fastest start of binder setting. Unfortunately, the increase in the CBPD component caused an increase in mortar swelling, which disqualified its use in the quantity of $> 25\%$. As for the HL component, its presence results in a substantial delay in the binder's start of setting, but the HL component also acts like a stabiliser of swelling caused by the CBPD. It is necessary to remember that the presence of lime also improves resistance to water of bound mixtures [9]. The analysis' complementation is the quality of matching of the response surface model parameters with the experiment results. The results are presented in table 4.

Table 4. Parameters of matching of the response surface model

Model parameter	Feature					
	Start of setting [s]	Change in volume [mm]	Bending strength after 7 days [MPa]	Bending strength after 28 days [MPa]	Compressive strength after 7 days [MPa]	Compressive strength after 28 days [MPa]
(A) CEM	191.27	10.47	6.46189	7.2864	34.448	46.4950
(B) $\text{Ca}(\text{OH})_2$	2901.23	-1.33	-0.01492	0.0956	0.247	0.3848
(C) CBPD	1480.97	86.58	0.34963	1.9017	1.525	3.8794
AB	-2013.33	146.77	-4.73014	-20.4994	-18.356	-57.0001
AC	-2312.35	67.11	3.59179	-8.9143	-2.300	-24.7821
BC	-6610.14	342.60	-3.17377	-16.0565	-1.806	-18.6292
ABC	-	-2099.53	-	140.6176	-111.009	109.9098
R^2	0.98	0.95	0.97	0.96	0.92	0.99
RMSE	166	6.6	0.32	0.41	0.86	1.3

When analysing the results of matching (table 4) the model with the experimental data, it is necessary to note that the results were characterised by a high determination coefficient R^2 . Furthermore, the Root Master Squared Error was relatively low in relation to the results of designation for the given feature. Nearly all model parameters had a substantial impact on the shape of the assumed response surface model (red colour). The occurrence of substantial interactions between the response surface model parameters proved the existence of synchronicity between the binder's components, which resulted, among others, in a decrease/increase in the mortar's volume. The high quality of matching the model with the experimental data favoured the optimisation with the use of the surface response model data. Furthermore, it is necessary to note that each component affected each of the standard properties differently. Due to the above, the binder composition optimisation in terms of its usefulness in mixtures bound using the deep recycling technology with foamed asphalt was justified. During optimisation based on the usefulness function model (2), the results for the normally setting and fast-setting binder were compiled in figure 4.

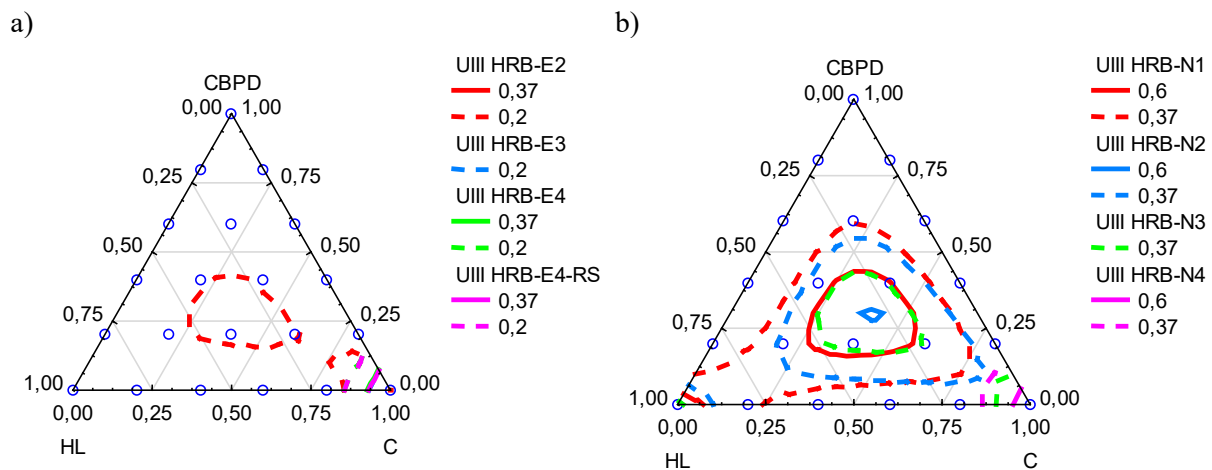


Figure 4. Results of optimisation in relation to standard requirements (table 3)

When analysing figure 4a, the most desired areas were located inside the solid lines according to the given type of binder ($UIII > 0.37$). The values inside the dotted lines ($UIII < 0.2$) represented unacceptable results. It is worth noting that the optimal solution aimed at obtaining a fast-setting binder was possible decisively for 100% of the CEM content. It was acceptable to use only 10% of additive of the HL and CBPD mixture. In the area of solutions for the HRB-E2 binder characterised by the lowest setting kinetics, there was a satisfactory solution around the area where the central point achieved a good value for the configuration $C/HL/CBPD = 0.4/0.4/0.2$. It was a result reflecting the solutions for a single response surface node. A decisive area of response surfaces are the $UIII < 0.2$ results, according to which it is not possible to create a fast-setting binder that features a component different than cement (C).

When analysing the optimisation results (figure 4b), it can be observed that satisfactory results were achieved inside areas marked with a dotted line ($UIII > 0.37$). Taking into consideration the requirements made for normally-setting binders, there was an area of acceptable solutions where the binder's usefulness results were deemed as satisfactory and good (solid line $UIII > 0.63$). In the case of the HRP-N1 binder, at least satisfactory results can be achieved for the cement content in the interval of C: 20÷75%, HL: 20÷75% and CBPD: 30÷75%, but it is necessary to remember that the percentage share of all components must amount to 100%. In the case of the HRB-N2 binder, satisfactory results were achieved for the area around the node representing the result of optimisation $C/HL/CBPD = 0.4/0.2/0.4$. For the HRB-N3 binder, satisfactory results were achieved for the response surface area described with the following component ranges: C: 25÷60%, HL: 25÷50% and UCPP: 15÷45%. For the HRB-N4 binder, the best results were obtained for the mixture of C:80% and 20% of other components. It is

necessary to note that the excessive quantity of cement substantially increases the binder's compressive strength after 28 days of curing, which disqualified its 100% share in the composition of a binder intended for bound mixtures. Thereby, the need to use components other than cement in the HRB-Nx normally-setting binder was justified.

5. Conclusions

The tests and analyses conducted with the use of a universal binder intended for mixtures recycled using the deep recycling technology with foamed asphalt provided the following conclusions:

- The use of each of the three components resulted in obtaining different properties of the universal binder intended for mixtures bound in the deep recycling technology. Due to the above, the optimisation process based on the generalised usefulness function was especially justified and recommended.
- The use of the CBPD additive caused an increase in the standard mortar's swelling, but also shortened the slurry's start of setting and increased the mortar's bending strength after 28 days of curing.
- The increase in the HL quantity caused a delay in the mortar's start of setting, but also improved the stability of the mortar's volume change, caused by large quantities of CBPD.
- The use of cement resulted in a quick binder setting, but also caused an excessive increase in the mortar's strength after 28 of curing, which disqualified its excessive quantity in terms of usefulness in unbound mixtures in the recycling technology.
- The occurrence of substantial interactions between the response surface model parameters proved the existence of synchronicity between the binder's components, which resulted in a decrease/increase in the mortar's volume.
- The observations included a small area of acceptable solutions for the fast-setting binders, which was met by the universal binder. On the other hand, there is a high probability of obtaining satisfactory results of this mixed binder in terms of the requirements for the normal-setting binder.

Acknowledgment(s)

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