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Modelling the effect of composition on the properties of gypsum concrete containing cenospheres

Introduction

Materials on the base of calcium sulphate have great promise for sustainable construction. In particular, the application of lightened gypsum concrete (LGC) to production of building partitions can give a considerable effect. To lower construction mass and expenditure of the binder introduced into mix are various light aggregates: perlite, vermiculite, sawdust, foam polystyrene granules, etc. Improving thermal properties of gypsum units these organic and inorganic aggregates increase water demand of the mix and decrease the strength of hardened material.

It is suggested that alumina-silica microspheres (cenospheres) formed as a part of fly ash [1, 2] could be used as efficient filler for lightened gypsum concrete. These particles (of size 10-350 microns) have unique combination of properties [2, 3]: low density and heat conductivity, spherical form, chemical inertness, high hardness and melting temperature. They find application in heat-insulating and sound absorbing cement based materials [3-5]. The use of waterproof cenospheres to replace (totally or partially) water absorbing aggregates in gypsum based composites could reduce the quantity of mixing water and the decrease in strength.

Experiment and models

When developing the lightened gypsum concrete varied in experiment were two groups of composition parameters shown in Table 1, corresponding to their values being the levels of normalised factors, $|x_i| \leq 1$. Microsphere size distribution with average about 0.1 mm and average size of perlite grains about 0.3-0.5 mm could suggest rather compact piling and possibility of forming relatively steady filler-aggregate skeleton.

Table 1
Values of composition parameters in the experiment

i	Factor X_i		Levels		
			$x_i = -1$	$x_i = 0$	$x_i = +1$
1	Aggregate factors – quantities (gypsum volume %) of:	cenospheres (CS)	30	50	70
2		perlite (P)	0	15	30
3	Matrix factors – dosages (% of gypsum mass) of:	superplasticiser (SP)	0.3	0. 5	0.7
4		air-entraining admixture (AE)	0.2	0.5	0.8

The ranges indicated have been chosen on the base of preceding trials. In particular, the lower limit of CS is conditioned by revealed growth of strength when introducing up to 20-30% of cenospheres. The required amount of water for each β-hemihydrate based composition was tried so that it would provide mix spread of 18 cm (at glass table).

Determined for 18 compositions, according to 4-factor 2nd order design of experiment [6], were water demand *W* (kg for 1 kg of dry mix), water-gypsum ratio *W/G*, the levels of material properties, including, density ρ (kg/m^3), compression and bending strength R_c and R_b (MPa), heat conductivity λ (W/m/K), and characteristic of soundproofing ability of the material R_S (dB). Analogous to index of noise-proofing for walls and partitions (defined by the norms used in Ukraine, equal to fall of sound pressure due to barring specimen) R_S can not numerically coincide with it but can serve as comparative criterion when developing soundproof materials. Not considered here are the data on setting time. As preceding trials have shown, initial setting time (between 7 and 21 min for 18 compositions) can be retarded with micro-dosages of citric acid without noticeable loss in strength. The experimental data presented in Fig. 1 shows the expected correlations of some properties, with *W/G* being between 0.57 and 0.68, at practically equal *W*=0.51-0.53 and amount of building gypsum 0.77-0.90 kg / kg of dry mix.

The data obtained have allowed the fields of material properties in four composition coordinates (of vector ***x***) to be described by non-linier 4-factor experimental-statistical (ES) models [7, 8], such as (1-2) for density and compression strength (at 1 and 6% experimental errors respectively, with significant coefficients at 10% risk).

$$\begin{aligned}\rho = 945.2 &- 39.5x_1 \pm 0\ x_1^2 + 6.5x_1x_2 \pm 0\ x_1x_3 \pm 0\ x_1x_4 \\ &- 73.3x_2 \pm 0\ x_2^2 + 5.6x_2x_3 - 8.7x_2x_4 \\ &- 5.5x_3 + 10.9x_3^2 + 7.5x_3x_4 \\ &- 9.8x_4 + 11.8x_4^2 \end{aligned} \qquad (1)$$

$$\begin{aligned}R_c = 5.46 &- 0.75x_1 \pm 0\ x_1^2 \pm 0\ x_1x_2 + 0.19x_1x_3 \pm 0\ x_1x_4 \\ &- 1.14x_2 \pm 0\ x_2^2 + 0.22x_2x_3 \pm 0\ x_2x_4 \\ &- 0.36x_3 + 0.40x_3^2 \pm 0\ x_3x_4 \\ &- 0.55x_4 \pm 0\ x_4^2 \end{aligned} \qquad (2)$$

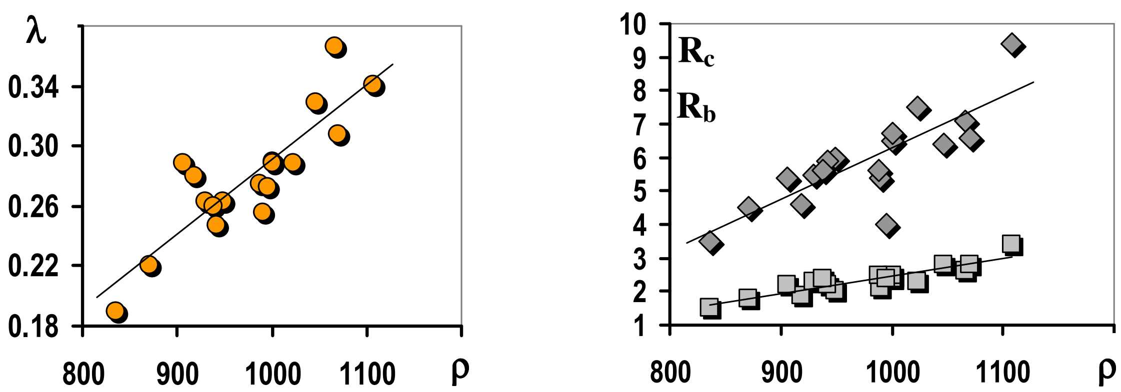

Figure 1
Scatter diagrams of experimental values of LGC properties

Analysis

With the models, maximal and minimal levels and other generalizing indices of the property fields have been determined. Here are some of them.

ρ_{min} = 830 kg/m^3 (at $x_1 = x_2 = +1$, $x_3 = -0.30$, $x_4 = 0.89$),

λ_{min} = 0.19 W/m/K (at $x_1 = x_2 = x_3 = x_4 = +1$),

$R_{c.max}$ = 9.1 and $R_{b.max}$ = 3.4 MPa (at $x_1 = x_2 = x_3 = x_4 = -1$),

$R_{S.max}$ = 42.5 Db (at $x_1 = 0.8$, $x_2 = x_3 = x_4 = +1$).

As it should be expected the desirable individual optima of the criteria belong to quite different compositions, quite "opposite" for λ and strength. So the need to find some compromise may arise when developing the material for specified conditions.

The individual influence of mix proportions on the properties is visualised with "quasi-one-factor curves" (Fig. 2) at minima and maxima zones (the curves are obtained by substituting the values of 3 other factors providing the minimum and maximum in corresponding model). The models and the curves show the governing effects of light filler-aggregate components on composite structure and rather ambiguous effects of matrix modifiers. The dosages of admixtures have proved to be more powerful factors for soundproofing index R_S increasing significantly with increase of SP and especially AE. Adding the cenospheres (from 30 up to 70%) would rise the level of R_S when it is relatively high (in zone of its maximum), but would lower it if the matrix, intergranular and interphase layers and the interfaces, are not "good" enough for this index (i.e., in zone of its minimum).

Different in direction for various properties the combined effects of the aggregate and matrix factors have been analysed with the help of diagrams "squares on square" and "cubes on segment", such as shown in Fig. 3 and in Fig. 4 for compression strength and heat conductivity.

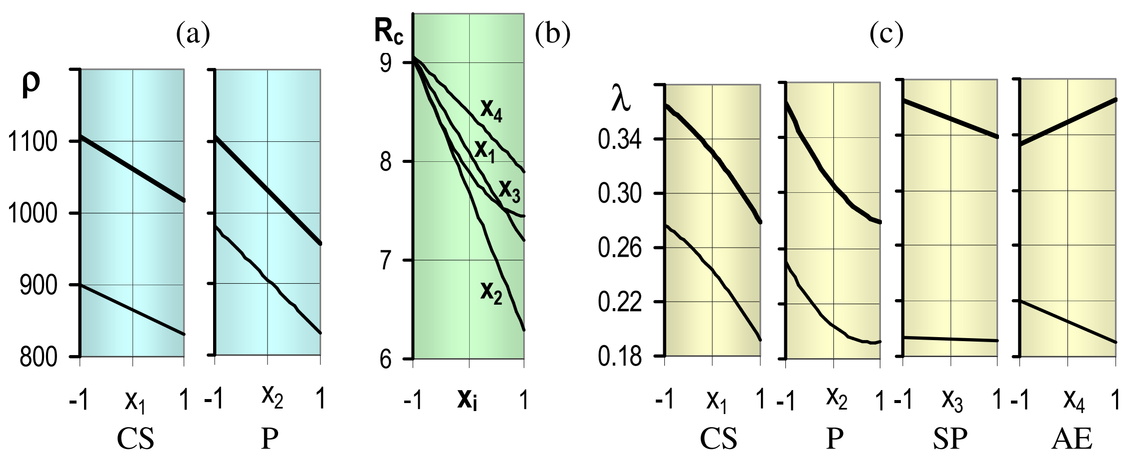

Figure 2
Effects of mix proportions on density and heat conductivity in zones of their maxima and minima (a, c), on compression strength in zone of its maximum (b)

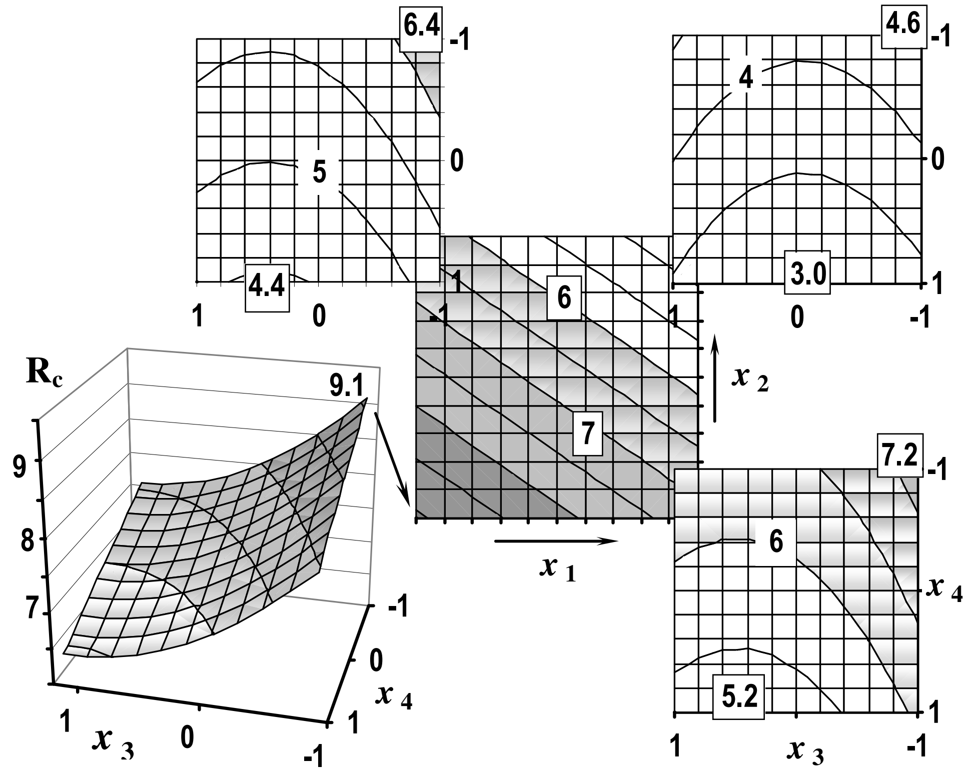

Figure 3
The fields of R_c in coordinates of SP and AE (i.e., x_3, x_4) at vertices (x_1, x_2 = ±1) of carrying CS+P square {x_1, x_2} and maximum of $R_c(x_3, x_4)$ in dependence of x_1, x_2

Possible increase in R_c and the maximum that could be achieved by controlling the dosages of admixtures presented on 4 diagrams in the vertices of central square (Fig. 3) come to 1.6-2.6 MPa and 4.6-9.1 MPa respectively and depend on light grains skeleton, the compositions with 30% of cenospheres without perlite ($x_1 = x_2 = -1$) providing the maximal increase and maximal strength. The isolines of $R_{c.max}$ over the region of SP

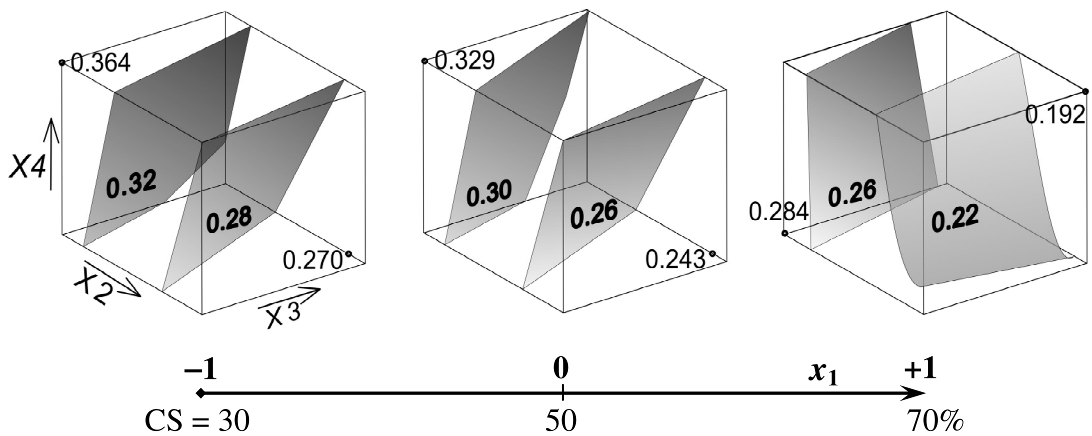

Figure 4
The fields λ(x_2, x_3, x_4) for compositions with various contents of cenospheres

and AE (over $\{x_3, x_4\}$-square) on the "carrying" square of CS and P show the decrease of this index when rising the content of light grains.

But this would improve heat insulation (lessen heat conductivity). The cubes in Fig. 4 confirm this fact with isosurfaces of λ. They demonstrate the changes in possibilities to regulate heat conductivity with P, SP, and AE when going from "low" to "high" content of cenospheres (in the range under study). With P being the most powerful factor, it could be noted that at low and median CS increasing the dosage of air-entraining admixture does not help to reduce λ.

Extracting latent information

The ES-models make it possible to extract the information latent in experimental data, which are convoluted in the models. In particular, the transformations of relations between structural and performance characteristics of mix and material with changes in technological conditions can be evaluated, with the help of computational experiments [8]. In this particular study correlation coefficients $r\{R_c, \rho\}= 0.81$ and $r\{R_b, \rho\}= 0.89$ by 18 pairs of experimental values (displayed in Fig. 1) have confirmed (at less than 1% risk) the significant positive correlation between density and strength. However these data could present the different populations (properties of compositions of essentially different structure). To reveal the existence or the absence of correlation and distinctions in relations between material properties in various formulation zones and to characterise these relations the statistical trials on local fields of the properties can be carried out [8, 9]. The paired samples of any size necessary for such analysis and for building possible prediction equations can be imitated in computational experiments with any number of generated compositions using the ES-models obtained.

Specifically, each of 2 diagrams in Fig. 5 presents the pairs of values (R_c, ρ) and (R_b, ρ) for 100 generated compositions with various quantities of admixtures (within the range $0.3 \le$ SP $\le 0.7\%$, $0.2 \le$ AE $\le 0.8\%$, $-1 \le x_3, x_4 \le +1$): **a** – at minimal CS = 30 and P = 0% ($x_1 = x_2 = -1$), **b** – at upper levels of CS = 70 and P = 30% ($x_1 = x_2 = +1$). It has turned out that the variations in dosages of admixtures show though significant but rather weak correlation of strength with the density, $r\{R_c, \rho\}= 0.32$ and $r\{R_b, \rho\}= 0.45$, when there are no light perlite grains in gypsum composite of rather "high" strength, containing optimal quantity of cenospheres. When the light skeleton comprises the substantial part of composite structure the relations become stronger, $r\{R_c, \rho\}= 0.67$, $r\{R_b, \rho\}= 0.72$; they can be evaluated and be used for predicting R by ρ.

Optimal compromise compositions

Presented below are the solutions of multicriterial optimisation problems, which could arise when designing the lightened gypsum concrete containing cenospheres. Two sets of the problems have been solved. For the first one the following conditions have been formulated: the density $\rho(\boldsymbol{x})$ and heat conductivity $\lambda(\boldsymbol{x})$ should be minimised and bending strength $R_b(\boldsymbol{x})$ should be maximised complying with specified requirements for

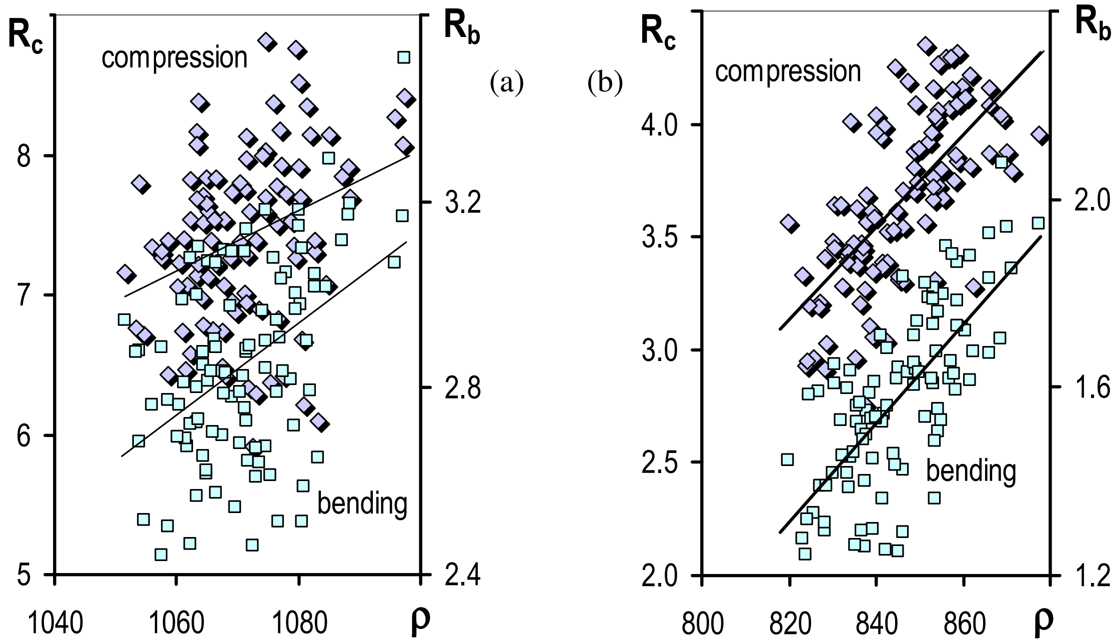

Figure 5
Scatter diagrams of simulated values of the properties for generated compositions with 30% of cenospheres without perlite (a) and with CS and P at upper levels (b)

compression strength, $R_c(\boldsymbol{x}) \geq R_{c.norm}$. The solutions have been found for several values of $R_{c.norm}$ specified for certain purpose materials: 5 MPa (required by Ukrainian norm for gypsum based materials for partitions), 4, 3.5, and 5.58 MPa (explained below).

To ensure the fulfilment of the requirements the values accounting for the error of corresponding ES-model should be added (with plus or minus) to specified property levels, defining the "guaranteeing" levels [8, 10] and thus more stringent requirements. In the particular case of $R_{c.norm}$ = 5 MPa the limiting value for R_c would become 5.58 MPa. This could be important when increased reliability is required.

It can be seen that some compromise between ρ_{min} and λ_{min}, on the one hand, and $R_{b.max}$ on the other must be found since the most "powerful" factors, CS and P, decrease all optimality criteria. Used to find the compromise has been the iterative random scanning of composition-property fields [10, 11]. At each of its iterations, firstly, the region of acceptable mixes is determined and than the ranges of compromise are narrowed in step-by step procedure.

The following results have been obtained, in particular, for $R_{c.norm}$ = 5 MPa (mix parameters being reasonably rounded):

- ♦ the content of cenospheres CS = 50% ($x_1 = 0$);
- ♦ the content of perlite P = 30% ($x_2 = +1$);
- ♦ the dosage of superplasticiser SP = 0.3% ($x_3 = -1$);
- ♦ the dosage of air-entraining admixture AE = 0.35% ($x_4 = -0.5$);
- • R_c = 5.1, R_b = 2.2 MPa, ρ = 899 kg/m^3, λ = 0.25 W/(m·K).

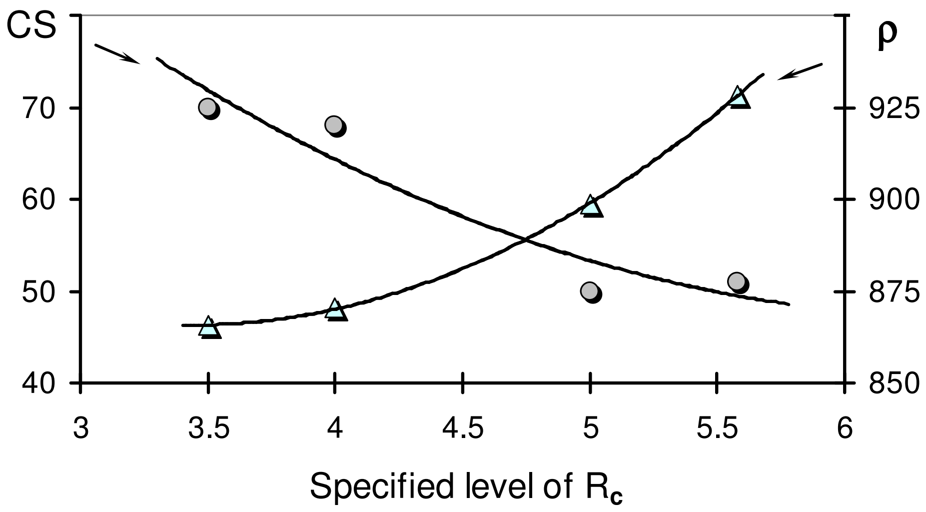

Figure 6
Quantity of cenospheres in optimal compromise composition and its density versus required minimal compression strength

Shown in Fig. 6 are the quantities of cenospheres in optimal compromise mixes found for 4 levels of $R_{c.norm}$ and the values of density of corresponding hardened composites. The growth of the latter with increasing $R_{c.norm}$ follows the quadratic dependency (determination coefficient equals 1). The greater is required strength, the less CS can be introduced. This relation is also described by quadratic function (Fig. 6), however linear approximation is also acceptable.

In the second set of optimisation problems one more optimality criterion has been added, soundproofing index R_S should be maximised. Here are the results obtained for $R_c \geq 5$ MPa:

- ♦ CS = 42% ($x_1 = -0.4$); ♦ P = 26% ($x_2 \approx 0.7$);
- ♦ SP = 0.46% ($x_3 = -0.2$); ♦ AE = 0.5% ($x_4 = 0$);
- • $R_c = 5.0$, $R_b = 2.0$ MPa, $\rho = 906$ kg/m^3, $\lambda = 0.27$ W/(m·K), $R_S = 37.5$ dB.

Conclusions

The promising lightened gypsum based composite containing cenospheres has been put forward and studied. The individual and combine effects of mix proportions on density, strength, heat conductivity, and soundproofing index have been evaluated and analysed with multifactor experimental-statistical models built on the data of designed experiment. The models have been used in computational experiments, which have enabled the information latent in experimental data on the relations between material properties and composition parameters to be extracted and optimal compromise compositions, sufficiently light, heat insulating, soundproofing, and complying with requirements for strength, to be found.

To make further progress there is a need for physical-chemical investigations of the interphase layers between gypsum matrix and cenospheres and other dispersed elements.

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