STRUCTURE CHARACTERISATION OF HIGHLY FILLED EPOXY POLYMER CONCRETES

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ABSTRACT
Highly filled particle composites, based on sand and epoxy binder, can be used as sound-absorbing materials. Experimentally determined densities may be employed in determining the content of the separate components, i.e. of sand, binder and pores.

KEYWORDS: polymer concretes, sand, epoxy binders

1. INTRODUCTION

Hard, porous, sound-absorbing materials are based on particle composites. The properties of the porous layer of bound particles depend on the porosity of the material, on the curvature of mutually interconnected air cells between the separate particles, and on the thickness of the material. A suitable binder is quartz glass sand, grain size 0.6 – 1.2 mm. The sand has sufficiently sharp and, as compared to mineral crushed material, also strong grains, it is non-flammable and, as compared to mineral crushed material, substantially less expensive and also more readily available.

Hard acoustic materials can be included in the group of polymer concretes together, e.g., with cast flooring. As opposed to the latter, however, the content of the binder in the composite must be very low, to avoid filling the empty spaces between the grains, so that the material will retain its high porosity. Moreover, the material must be sufficiently strong and resistant to change of shape when heated. This is highly demanding particularly as regards the binder used. Binders based on epoxy resins meet these demanding requirements best (Žikovský, Kolář, 2000).

In this paper, we shall deal with the characterisation of particle composites in terms of volume fractions of the separate components, i.e. sand, binder and pores. These parameters can be calculated from experimentally determined densities. The results may be employed in product quality control, etc.

2. EXPERIMENTAL

RAW MATERIALS USED

Quartz glass sand from the locality Střeleč close Turnov, grain size 0.6 – 1.2 mm. The results of screening are given in Table 1.

CHS Epoxy 510 (epoxy group content 5.1 mol/kg,).
Telalit 410, (220 g of curing agent/1 kg epoxy resin), (Spolchemie CZ).

PREPARATION OF SAMPLES

Samples of composites, based on sand, with different contents of epoxy binder were prepared in the shape of plates, dimensions 300 by 300 by 5 mm. The surface of the steel mould was treated by GT85 Teflon separator. The epoxy resin was mixed with the curing agent in a mass ratio of 100:22. The required

Table 1 Properties of quartz sand

Grain size[mm]	Mass %
2-3	0
1-2	21
0.5-1	78
0.2-0.5	1
0.1-0.2	0

amount of binder was then mixed with dry sand. After homogenisation, the mould was filled with the mix, and the surface of the plate was rammed and rolled. The curing took place at a temperature of 100 $^\circ$C for a period of 30 mins.

DENSITY MEASUREMENTS

The actual density of the sand and cured epoxy resin was determined by pycnometer (Brož, 1974). The actual density of the composite samples was calculated by adding the densities of both solid components. The bulk density ρ_B and the apparent density ρ_B of the composites were determined using a modified hydrostatic method based on three weighings – dry sample in air (G_0), sample fully

saturated with distilled water in water (G_1), and after being removed from water (G_2) (Kolář, Svítilová 1997).

$$\rho_B = \rho_l \frac{G_0}{G_2 - G_1} \qquad (1)$$

$$\rho_A = \rho_l \frac{G_0}{G_0 - G_1} \qquad (2)$$

where ρ_l is the density of the fluid used (in this case distilled water).

3.   RESULTS AND DISCUSSION

The samples were characterised by mass and volume fractions of the separate components. The mass fractions of sand and epoxy resin were calculated from the raw material charges. The volume fractions of all components, i.e. of sand, epoxy binder, closed and open pores were calculated from experimentally determined densities of the composite, sand and cured epoxy resin.

The _bulk density_ ρ_B includes the whole volume of the body, inclusive of the gaseous filling of cavities, if any. The apparent density ρ_A includes all solid phases and closed pores in the body volume, or more accurately, the pores not accessible to the fluid in question. The _real density_ ρ concerns the solid phases of the body only. If the body does not contain closed pores, then, of course, $\rho_A = \rho$.

The actual density of the composites was calculated based on the additivity of both solid components – the filler and the binder:

$$\rho = \left(\frac{W_E}{\rho_E} + \frac{W_S}{\rho_S} \right)^{-1} \qquad (3)$$

where W_E and W_S are the mass fraction of epoxy and sand, ρ_E and ρ_S are the corresponding real densities, determined by pycnometer.

The bulk, apparent and real densities were used to calculate the volume fractions of all components of the composite:

Open porosity

$$v_O = 1 - \frac{\rho_B}{\rho_A} \qquad (4)$$

Closed porosity

$$v_C = \rho_B \left(\frac{1}{\rho_A} - \frac{1}{\rho} \right) \qquad (5)$$

Epoxy Binder volume fraction

$$v_E = W_E \frac{\rho_B}{\rho_E} \qquad (6)$$

Sand volume fraction

$$v_S = (1 - W_E) \frac{\rho_B}{\rho_S} \qquad (7)$$

The results of the measurements and given in Tab. 2. The open porosity of the composites is given mostly by the amount of binder in the composite. As opposed to this, the magnitude of the closed porosity depends, to a considerable extent, on the preparation process. The closed pores consist mainly of air bubbles, which are created when the initial composition is mixed. If the bubbles cannot be removed before the binder turns to gel, they become fixed in the hardened resin. Since the transport of randomly created bubbles depends on the thickness of the later, it is not surprising that closed porosity appears only under high binder content, about from mass fraction $W_E = 0.11$, see Fig. 1.

As assumed, the volume fraction of the epoxy binder increases with the content of the binder, practically linearly. As opposed to this, the volume fraction of sand (up to a binder content of $W_E = 0.09$) changes only little. It follows that also the aggregate volume of the binder and all pores $(v_E + v_O + v_C)$ will be roughly constant within the said interval. Thus, the binder gradually fills the cavities in the sand without the total volume of the body increasing proportionally. These facts are evidenced in Fig. 2, in which the dependence of v_S, $(v_E + v_O + v_C)$ and of the relative changes of the total volume on the amount of binder in the composite are plotted.

If the volume of the body is considered constant, which is satisfied if the mass fraction of the binder in the mix does not exceed 10 mass %, it is possible to estimate the bulk density of the composite, which for a given type of sand depends only on the binder content, relatively well:

$$\rho_B = \frac{\rho_S v_S}{1 - W_E} \qquad \text{for } v_S = const. \qquad (8)$$

However, the apparent density depends more in the number of closed pores in the binder. This depends on the manner in which the composite is prepared, and cannot be foreseen very well. In subsequent deliberations, we shall consider the ideal case, in which the material contains no closed pores and, of course, $\rho_A = \rho$.

The bulk density increases with binder content, however, only up to a value corresponding to the complete filling of all open pores and cavities $(v_0 = 0)$. This is where ρ_B reaches its maximum value, for which:

$$\rho_B = \rho = \rho_E (1 - v_S) + \rho_S v_S \qquad (9)$$

The binder mass fraction, at which the porous system becomes filled ($v_O = 0$), is calculated from the relation:

Table 2 Densities, mass and volume fractions of studied composites

W_E	ρ [g cm^{-3}]	ρ_A [g cm^{-3}]	ρ_B [g cm^{-3}]	v_S	v_E	v_O	v_C
0	2.643[*)]	-	-	-	-	-	-
0.01	2.606	2.593	1.832	0.685	0.018	0.294	0.000
0.02	2.571	2.570	1.878	0.694	0.037	0.269	0.000
0.03	2.549	2.541	1.883	0.691	0.048	0.259	0.002
0.04	2.532	2.524	1.855	0.676	0.056	0.265	0.002
0.04	2.515	2.520	1.886	0.684	0.066	0.252	0.000
0.05	2.499	2.483	1.880	0.677	0.075	0.243	0.005
0.05	2.483	2.475	1.915	0.686	0.085	0.226	0.003
0.06	2.467	2.440	1.922	0.684	0.095	0.212	0.009
0.06	2.452	2.460	1.970	0.697	0.106	0.199	0.000
0.07	2.437	2.438	1.939	0.682	0.113	0.205	0.000
0.08	2.422	2.426	1.963	0.687	0.124	0.191	0.000
0.09	2.380	2.370	1.946	0.669	0.148	0.179	0.004
0.11	2.329	2.337	1.950	0.656	0.182	0.166	0.000
0.16	2.218	2.117	2.032	0.647	0.269	0.040	0.044
0.20	2.126	1.983	1.944	0.588	0.326	0.019	0.066
1.00	1.193[**)]						

[*)] Pycnometric density of sand   [**)] Pycnometric density of cured epoxy resin
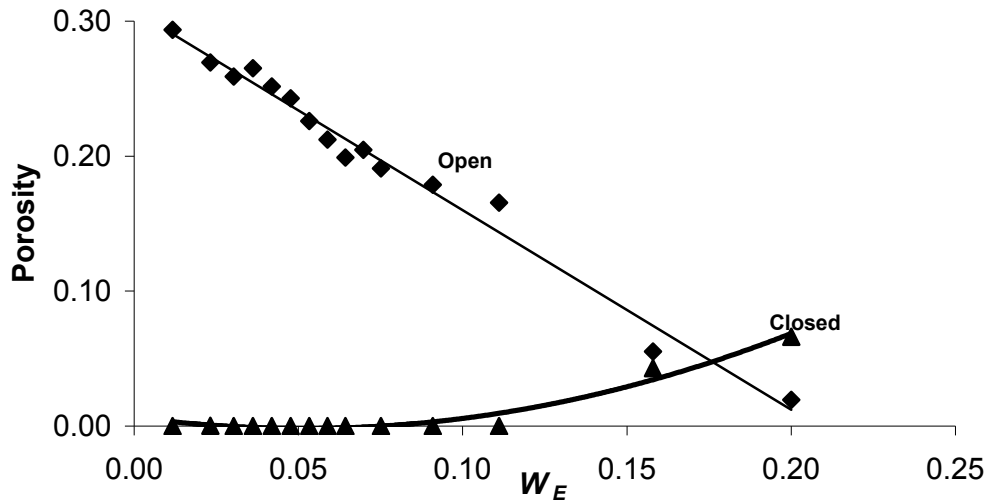
Fig. 1   Dependence of open and close porosities on epoxy binder fraction
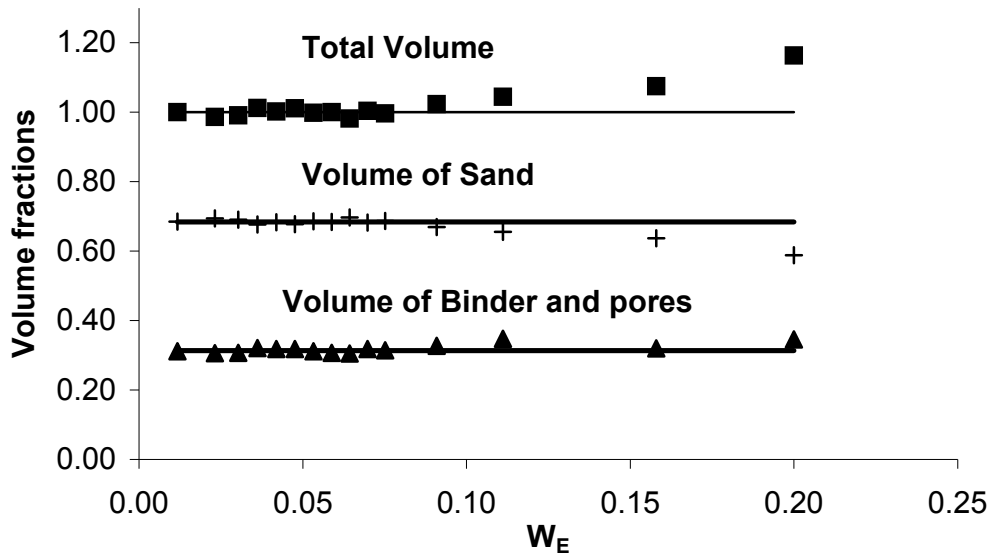
Fig. 2   Volume fractions of composite components vs. weight fraction of epoxy binder
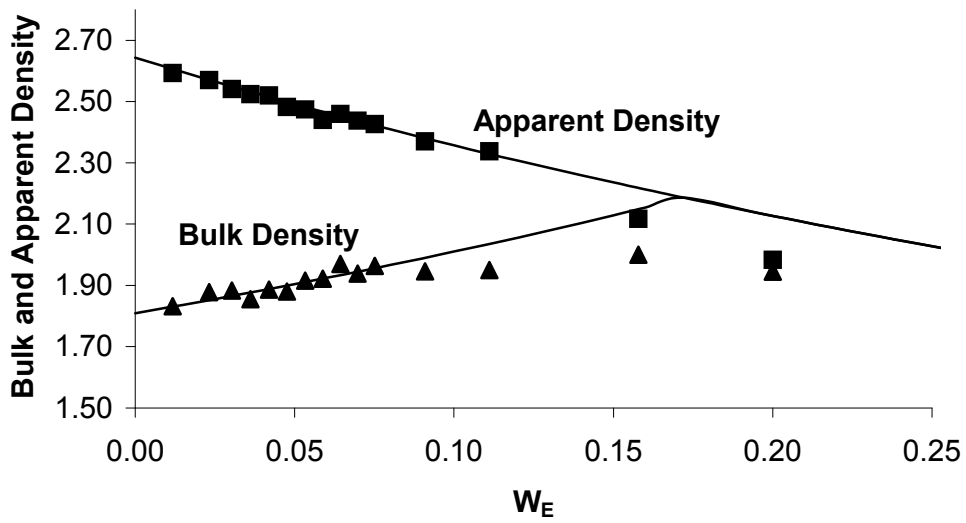

Fig. 3   Dependence of Apparent and Bulk densities on weight fraction of epoxy binder
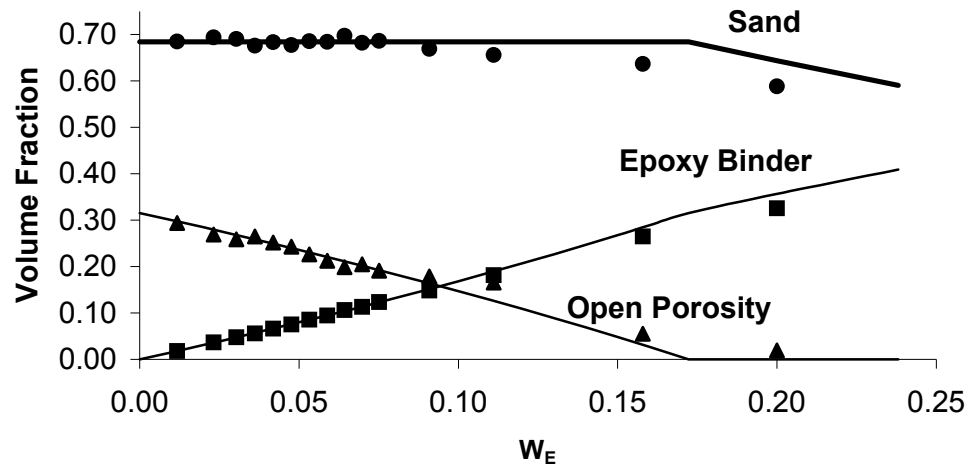
Fig. 4   Volume fractions of components

$$W_E^{'} = \frac{(1-v_S)\rho_E}{\rho_E(1-v_S)+\rho_S v_S} \qquad (10)$$

When this limiting value, W'_E, is exceeded, the composite volume continues to increase proportionally to the binder content. There are no opened pores in the body, and the bulk, apparent and actual densities area equal:

$$\rho_B = \rho_A = \rho \qquad (11)$$

The experimentally determined apparent and bulk densities are fitted in Fig. 3 to the functions calculated from Eqs (3) and (8).

Based on the calculated densities, the porosity and volume fractions of the other composite components can be estimated for the given type of sand from the relations given above. The dependence of open porosity and volume fractions of both solid phases on the mass fraction of the binder is depicted in Fig. 4. The relations derived on the assumption of constant volume are fitted to the experimental points in this figure. This indicates that the said relations can be used to estimate the structural characteristics of the composite, understandably only within the scope of the validity of the assumption that the volume is constant, i.e. in the interval $W_E \in (0,0.1\rangle$.

4.   CONCLUSION

The above relations can be employed in preparing particle composites with a given porosity, or to determine the material characteristics of composites, e.g., in product quality control. Based on the experimentally determined densities ρ_A and ρ_B, we can calculate the volume fractions of both solid components and the porosity from Eqs (5), (7) and (8). As demonstrated above, given the considered binder content (up to 10 mass %) all the pores in the composite are open, hence, it holds that $\rho_A = \rho$. Under these conditions, the mass fraction of the binder can be calculated from Eq. (4). With samples of poor quality, we are able determine retroactively breaches of technology, if any, e.g., lower content of binder, or imperfect compacting of the mix, which is reflected in higher porosity.

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