

THE RESEARCH OF THE WETHER RESISTANT GYPSUM-ASH-ALKALINE ARBOLIT STRUCTURE BY SCANNING ELECTRON MICROSCOPY

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Abstract. *The article gives results of the study of structural and mechanical characteristics of wether resistant gypsum-ash-alkaline arbolit that is relevant to the laws of strength, alignment and congruence by means of SEM .*

The quality of wether resistant gypsum-ash-alkaline arbolit depends on structural mechanical characteristics of the composite and first of all on the used filler and its dispersion.

As a filler the local plant raw material is used in work that provide a composite struture of arbolit which corresponds to the laws of strength, alignment and congruence.

The experimental theoretical research of the arbolit structure based on the plant biding composition (PBC) and modified polymer-silicate-gypsum binding material (PSGB) includes:

- research of adgesia and contact zone in the arbolit structure;
- impact of dispersion of the organic filler on its strength characteristics with account of level of the filler and fraction content of the uncemented composite.

Using the stereometrical technique of structure assessment defines prospectiveness and purposfulness of producing new composite from PBC and PSGB without using cement.

The structure of the arbolit filler was done by means of optical microscopes МБС-10, МУ-2, and also long-focused microscope made to take photos of microfilms.

The samples were cut out of arbolit in the form of cubes sized 10x10 mm and 20x20 mm. Two faces are upper and lower planes and 4 other faces are in the plane of the arbolit cross section.

As the arbolit structure is asinotropic, flatly oriented parallell to the sample plane to detect the geographical location and volume part of constituents it is enough to study only longitudinal and cross sections [1].

The sections for microscopical examination were prepared after their treting with paraffin to color and shift the particles.

The section microphoto treated with paraffin is shown in picture 1.

To detect the volume parts of constituents we used point-counting technique of A. A. Glagolev [1] by analogy with metalography but with account of asinotropy of particles. The choice of this technique is conditioned by the fact that it is the most convenient and available to count in the sight view of microscope with the help of eyepiece with a grid.



Fig.1. The microphoto of the section treated with paraffin along the filler particles orientation (magnified by 300 times).

Magnification (the microscope lens) was chosen so that the particles size of the constituents were not smaller than the distance between the grid knots. The scheme to detect the volume parts of the filler phases by point counting technique with overlaying the square grid with 25 knot points is shown in picture 2.

As the number of the structural constituents was not more than two we counted the number of knot points marked on every constituent apart. These numbers referred to their sum show the volume part of every structural constituent of the filler.

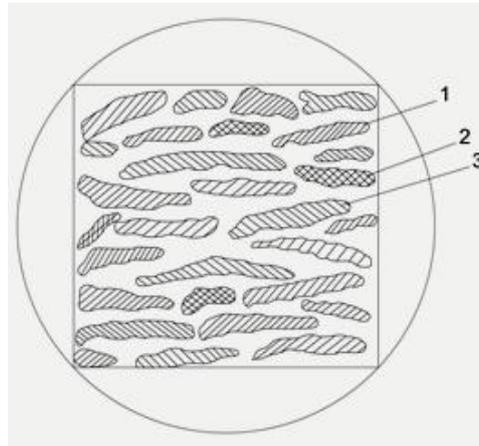


Fig.2. The scheme of detecting the volume parts of the filler phases by a point counting technique: 1,2- the particles of the different fraction straw; 3- the smallest fraction of particles.

The reliability of the received result of the point counting analysis is conditioned by the total number of the used points and depend on the volume part of the structural constituent in the composite. The value of absolute error ε depends on the measure number and volume part as follows:

$$\varepsilon = t \sqrt{\frac{\sum V(1 - \sum V)}{z}} \quad (1)$$

where εV – a volume part of this constituent; z – number of measuring (points number); t – the standard deviation varying depending on the required estimated probability [1].

To increase the accuracy of measuring we counted in several sight views (6-60) on one section and on several sections of the same sample and then averaged the result for every sample and counted the standard deviation:

$$\sigma = \sqrt{\frac{\sum_{i=1}^z 1(\Delta V_i)^2}{Z(Z-1)}} \quad (2)$$

where z – number of measuring; $\Delta V_i = |V_i - V|$ - deviation of the volume part at i th measuring from the average volume.

To gain the unknown before information about the character of breakage and the structure of the arbolit fracture surface we used the scanning electron microscope (SEM) make B – 301 magnification to 20000, resolution 15 nm (150 \AA).

The samples examination was done at accelerarating voltage 25 kVt. The resolution values obtained by means of SEM while examining the breakage surface allowed receiving a clear image of the breakage processes mechanism and structural character of complicated phases of the filler of plant material [2].

The principle of scanning electron microscopy is based on scanning the electron ray in the form of electron bunch (prober), the ray with diameter 10 nm synchronously transmits the signal to the kinescope on the sample surface point by point. When the electron ray gets any point of the sample the secondary or reflected electrons are knocked out of the material. The image brightness of the oint on the screen depends on number of the electron output. High output of electrons of the material results a

light image point on the screen, low output corresponds to a dark point. In the interval between these values of electron «outputs» we can observe grey points of different shades. The first ray (probe) is formed in a vacuum column (electron gun) of the scanning electron microscope. The electrons leave the heated cathode and are accelerated by the electrical field of voltage 1-50 kVt, with the cathode acceleration the ray is focused by three electro-magnet condenser lenses and with the help of the bending coils are scanned by analogy to receive the most objective result of the sample characteristics.

To receive more objective information about the initial including the structural condition of the studied samples –plates made of plant material they, as a rule, are left without purification that is the same way as they are supplied for examination. However, not all material is for the immediate examination with SEM. As the researched sample must be electroconductive the non-conductive materials are covered with a firm fats layer of silver or other metal (advisably at metal spraying) before examination. To examine in the scanning electron microscope the fractures of the reserached samples sized 10x20 mm were sprayed with a silver layer 50 A, that is the layer was almost invisible in the microscope and did not influenec the general idea of the researched struture. During the industrial examination of such mayerials it is very important to get an even distribution of the filler's particles along all thickness of the sample to receive the maximum contact of the particles and bast fibre. The analogs of the plates fracture surfaces and quite homogenous structure are shown in picture 3.

The fractographic analysis by means of SEM allows to establish the main peculiarities of the arbolit microstructure made of plant material that was not studied before.

The total number of the used points is $60 \times 20 = 1200$. 707 points got on the 1st structural constituent – straw fraction 10/7. Hence, the sought volume part of this constituent is $707:1200 = 0,83$ or 83,3 % by the filler's volume. 130 points got on the 2nd structural constituent (fracture 10/5), consequently the sought volume part of this constituent is $130:1200 = 0,10$ or 10% by volume. 84 points got on the 3d structural constituent. So, the sought volume part of this constituent is $84:1200 = 0,07$ or 7% by volume.



Fig.3. The surface of the arbolit fracture of density 600 kg/m^3 silver treated (magnified by 300times).

According to pictures 3 and 4 the section structure is examined in the sight view using the eyepiece grid with 25 knot points.

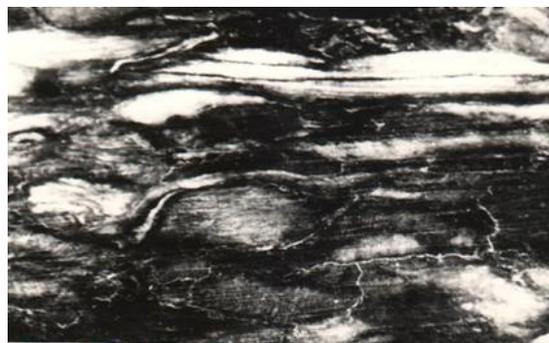


Fig.4. The section microphoto along the particles orientation of the filler (magnified by 300times with fracture content: 1-82%; 2 – 12%; 3 – 6%)

If $T = 0,674$ according the data [1], we will receive by the formula (3.1) the possible absolute error of the analysis expressed in parts of the filler volume (or the section square):

$$E = 0,674 \times 0,83 \times (1 - 0,83) / 1200 = 0,0079;$$

$$E = 0,674 \times 0,10 \times (1 - 0,10) / 1200 = 0,0051;$$

$$E = 0,674 \times 0,07 \times (1 - 0,07) / 1200 = 0,0037;$$

Hence, the true volume part of the first structural constituent is in the limits 0,83-0,0079 with estimated probability 0,5 .

The volume part of the second structural constituent is in the limits of 0,10 – 0,0075 with estimated probability 0,4. The relative error is 3,2 %. The volume part of the third structural constituent is in the limits of 0,07+- 0,0037 with estimated probability 0,5. The relative error is 5,1 %.

The sections microphotos of the plate samples of different volume, the filler's constituents where every type is colored brightly and treated with paraffin along orientation of the filler's particles and also in the cross section are shown in pictures 6 and 7.

In picture 6 of the sample longitudinal section of the plate all constituents- particles 1,2,3 are colored in different colors and are clearly seen in the microphoto of the plate section structure.

In picture 7 you can see the section microphoto in the cross section of the sample where the parts of the structural constituents are observed in their volume relation and their homogenous orientation and preseed character. Thus, using modern optical equipment to take photos of microsections with three structural constituents of the filler's composite material we detected the optimal volume parts of every constituent in the composite structure.



Fig.6. The section microphoto of the arbolit sample with every filler type colored in the cross section

The fractographic analysis of the samples in the electron microscope showed that particles of the examined arbolit samples both at fracture and surface section are homogeneously distributed in the arbolit structure and show their good mixture capacity with the binding element in the conglomerate.



Fig.7. The surface of the arbolit sample «fracture» with particles oriented in the longitudinal direction (magnified by 1000times)

In pictures 8 and 9 we can clearly observe the even and firm connection of particles to each other including a binding element providing even adhesion to the filler surface.

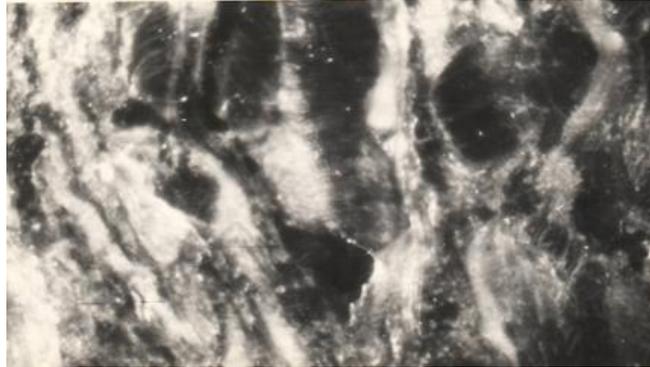


Fig.8. The surface of the arbolit sample «fracture» $P=650 \text{ kg/m}^3$ with particles oriented in lateral direction (magnified by 1000times)



Fig.9. The surface of the arbolit sample «section» with small particles – balls (magnified by 1000 times)

Thus, the electron microscopical examination allowed establishing the distinctive peculiarities of the goods structure made of gypsum-ash-alkaline binding ones with fillers by a standard pressing method of production. Using the method of pressing at low pressure of plastic mixtures (to 10 MPa) let us get a dense, block and strong structure of the gypsum-ash-alkaline stout.

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