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## STRUCTURE AND TEXTURE FORMATION OF FROZEN SAPROPEL

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Based on lab modelling and analyses of the processes of structural and textural formation in frozen high humid lake organogenous sediments has been carried out. Experimental data shows, that the process of freezing of organogenous high humid grounds causes humidity transfer from this zone into the frozen one, and its local redistribution in the boundaries of freezing and frozen zones appearing to extend differently depending on the conditions of heat exchange. In conditions where low intensive heat exchange prevails, humidity transfers from the thawed zone of organogenous ground into the frozen one with the subsequent freezing of migrated water presenting segregational layers of ice. Local redistribution of water mainly appears in conditions of intensive heat exchange at a high speed of freezing and is the result of expression and subsequent freezing of the connected water during the process of dehydration of the organomineral frame and of coagulation of the organic components of the rock.

### INTRODUCTION

Exploitation of lacustrine organogenic sapropel sediment as a valuable natural raw material, working all year round through technologies of their recovery in areas with stable winter freezing is impossible without taking into account the mass-exchange, physico-chemical and texture formation process caused by cryogenic phenomena. The efficiency of the existing technology of de-watering sapropel by way of freezing in precipitation tanks depends to a significant extent on the character of transformation of the texture of sapropel as a result of freezing. Unlike mineral soils, the processes which occur in frozen sapropel have not been thoroughly studied. A series of problems need further study. The most important of them are the following: study of the mechanisms and regularities of cryogenic texture- and structural formation, discovery of qualitative and quantitative relations of thermal and mass-exchange and cryogenic texture- and structural formation, predicting changes of structure and properties during freezing, and choosing the optimal freezing modes.

In accordance with the above mentioned the aim of this study was the research of formation and transformation of cryogenic macro- and microtexture of sapropel in a vast range of thermodynamic conditions of freezing, taking into account the mass-exchange, physico-chemical and physico-mechanical processes on the basis of laboratory modelling.

### THE TECHNIQUES OF RESEARCH

Modelling of thermal- and mass- transfer processes, cryogenic texture- and structural formation in sapropel was conducted on the basis of use of laboratory equipment and instruments. The main feature of this set was a laboratory installation (Brovka, Murashko, 1989). Its construction ensures the maintenance of a one-dimension temperature field during the freezing of ground

samples with a diameter of 140 mm and 300 mm in height. The installation allowed us to conduct a comprehensive study of thermal- and mass-transfer parameters in the freezing dispersal systems with automatic changing and regularity of temperature, and alteration of swelling degree.

The experiments were conducted on the sapropel samples of the upper- quaternary age with irregular composition patterns, different initial water content which did not undergo seasonal freezing in natural conditions. Sapropel of different compositions have been studied during modelling: organic, carbonate, siliceous, mixed- collected from the deposits of Byelorussia (Table 1).

Table 1. Characteristics of the studied sapropel of natural composition

Type of ground	Density of ground $\gamma$ , g/cm <sup>3</sup>	Density of matrix (skeleton) $\gamma_f$ , g/cm <sup>3</sup>	Natural humidity W, %	Ash content A <sup>c</sup> , %
Organic sapropel	1.03	0.042	2000	12.7
Carbonate sapropel	1.22	0.49	142-183	84.0
Siliceous sapropel	1.1	0.39	379	75.8
Mixed sapropel	1.08	0.47	340	57.0

Sapropel samples were frozen at given boundary conditions to the depth of 100 mm. After one-side freezing of samples they were cut in slices and the humidity and density were

determined. The results were used in drawing a diagram of curves reflecting the pattern of water distribution along the height of the samples at different time intervals. Thermal- and mass-transfer parameters were calculated. Cryogenic macro- and microstructures were studied in samples of organogenic soil prepared by means of vacuum sublimation, in replicas and thin sections with the use of optical and electron microscopy (Ershov, 1988). The universal digital system of image analysis IBAS-2000 produced by "Opton" which was capable to conduct various measurements, transformation and classification of the studied objects was used in order to perform comprehensive analysis of the processes of texture- and structure formation depending on the parameters of heat and mass transfer.

Application of the method of image analysis for research of the cryogenic structure of frozen organogenic soils enabled us to calculate a series of object- and field specific textures and structural parameters which reflect the form, dimensions, orientation in space, location with respect to one another, and pattern of distribution of ice and organomineral constituents. The following object-specific parameters were calculated: area (S), perimeter (P), maximal and minimal diameter of ground particles ( $D_{max}$ ,  $D_{min}$ ), their orientation ( $X$ ,  $Y$ ,  $\psi$ ) and form coefficients ( $K_f$ ,  $K_{el}$ ). Determination of field specific parameters with respect to the ice constituent allowed the calculation of voluminous schlieren ice content in the rock  $i_t$  and, separately, the ice content due to horizontal and vertical schlieren of ice. The application of the image analysis system let us obtain the data on water content of aggregates of organomineral matrix of soil  $W_{ra}$ . The volume distribution pattern ( $W_{vol}$ ) down the frozen zone of samples was determined layer-by-layer by means of thermostatic drying. Afterwards, ice content was calculated with the image analyser according to the image of the cryogenic structure obtained from the replica. The density of organomineral layers was calculated as:

$$W_{ga} = \frac{W_{vol} - i_t}{1 - i_t}$$

The mentioned parameters helped to establish the quantitative relationship of processes of re-distribution of water and formation of cryogenic macro- and microstructure of freezing sapropels.

#### THE RESULTS AND THEIR ANALYSIS

The result of experiments on one-sided freezing show that the physical and chemical processes in sapropels depends a lot on the availability of organic components of the rock matrix. It stimulates the coagulation processes during freezing, influences the water transfer mechanism and cryogenic texture and structural formation on the whole. Despite the anomalously high natural water content in sapropels, the character of re-distribution of water in them is similar as in mineral soils. Ice was also observed here in the frozen zone as well as the dehydration in the thawed zone which is due to the gradient of general thermodynamic potential causing the flow of water from the thawed zone to the frozen one. The dynamics of freezing is characterised by the most significant water re-distribution in conditions of low-intensity thermal exchange at low

velocities as the velocity of the division boundary between the phases, decrease of temperature gradient in the frozen zone and the density of migrating flow of water (Fig.1).

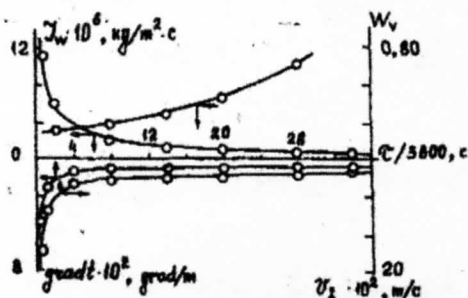


Figure 1. The character of the density of water migration flow ( $J_w$ ), temperature gradient ( $\text{grad } t$ ), humidity ( $W_{vol}$ ) and velocity of freezing ( $V_{fr}$ ) temporal changes in the carbonate sapropel  $W_{vol}=0.71$ ;  $\gamma_f^0=0.51 \text{ g/cm}^3$ ,  $t_0=-3.4^\circ\text{C}$ .

The results of experimental studies confirm that texture- and structural formation during freezing of sapropel occurs in conditions of two mechanisms of mass-transfer: migration of water from the thawed zone of the freezing ground into the frozen one with subsequent freezing in the form of layers consisting of segregation ice (migration-segregation mechanism) and local re-distribution of water within the freezing and frozen zones during the process of driving off combined water and its subsequent freezing with the dehydration of organomineral aggregates and coagulation of organic components (coagulation mechanism). Cryogenic texture- and structural formation in freezing sapropel occurs in several stages in correspondence with the alteration of the prevailing mechanism of mass transfer during freezing.

At the initial stage of freezing which is characterised by high values of  $\text{grad } t$ , the process of texture formation is regulated by the laws of free water crystallization which corresponds to the prototrope stage of crystallization. At the following stage the forming ice matrix promotes oriented growth of ice crystals which constitutes vertical schlieren. They thicken with depth and pull the associates of organomineral matrix apart while deforming and consolidating it. The process of consolidating the sapropel matrixes accompanied by dehydration with driving the combined water off which freezes at the periphery of ground aggregates. Dewatering of the matrix causes activation of the process of aggregation and coagulation of organomineral component. Height contents of the organic component in the sapropel which possesses high adhesion characteristics, promotes combining material particles into organo mineral aggregates. Their formation is accompanied by the nearing of particles and the increase of their surface and it causes still more deep dewatering of the organomineral matrix. The vertical ice schlieren were formed during this process which is due to the driving off and subsequent freezing of combined water during dehydration of organo-mineral matrix and coagulation of the organic component of the rock (coagulation-segregation mechanism of ice formation). As the front goes forward and the rate of freezing beside the coagulation-segregation mechanism of ice formation, the role of migration-segregation factor increases which

leads to the occurrence of horizontal schlieren of ice of second generation and while having the higher importance is the lower rate of freezing.

Relation between the mechanism of mass transfer and ice formation is determined by the conditions of freezing (Fig.2) and the composition of organogenic soil. The rate of freezing is higher, temperature gradient in the frozen zone and lower the ash content of sapropel, the more intense is the coagulation-segregation mechanism and the lesser role belongs to the migration-segregation mechanism. The influence of the altering rate of freezing  $V_{fr}$  and the temperature gradient in the frozen zone  $grad t$  on the processes of texture- and structural formation can be seen in the example of carbonate sapropel (initial humidity  $W_0=183\%$ ,  $\gamma_f=0.42 \text{ g/cm}^3$ ) frozen in a closed system with changing thermostat temperatures of the surface of the sample from  $t_0=-2.1^\circ\text{C}$  to  $t_0=-9.9^\circ\text{C}$ . The laboratory modelling has shown that total ice content due to schlieren ice formation regularly decreases as the  $grad t$  increases in the frozen zone. The effect of horizontal  $i_h$  and vertical  $i_v$  components of the schlieren ice formation on changing total ice content are not equal and also depends on  $grad t$  values in the frozen sapropel zone. At  $grad t$  values below  $1.5 \text{ grad/cm}$  the schlieren ice formation sharply increases due to segregation layers  $i_h$  while  $i_v$  features the opposite tendency. Increase of temperature gradient up to  $grad t > 1.5 \text{ grad/cm}$  leads to monotonous augmenting of the contribution of the vertical due to segregation. At the values of  $grad t$  exceeding  $12 \text{ grad/cm}$  the ice content caused by the vertical layers becomes predominant (Fig.2). Temperature of thermostat conditions influences the distribution of components of schlieren ice formation to the depth of the sample while tracing the zone of changing the prevailing mechanism of mass transfer (Fig.3).

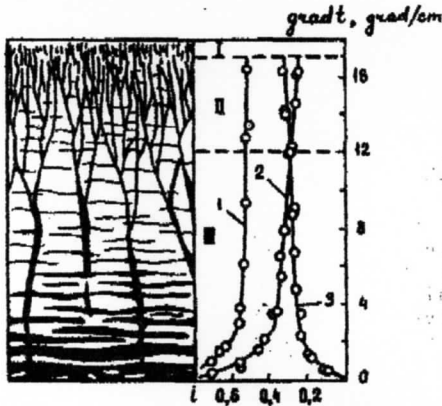


Figure 2. The character of the cryogenic texture, schlieren ice content in the rocks component and the prevailing mechanism of ice segregation changes due to the freezing conditions of the carbonate sapropel ( $W_0=183\%$ ,  $\gamma_f=0.42 \text{ g/cm}^3$ ):

- I - the zone of prototrope crystallization;
- II - the zone of prevailing coagulation-segregation ice formation;
- III - the zone of prevailing migration-segregation ice formation;
- 1) - the total schlieren ice content;
- 2) - the ice content due to horizontal schlieren;
- 3) - the ice content due to vertical schlieren.

Total structural ice content and ice caused by the horizontal schlieren increases with the

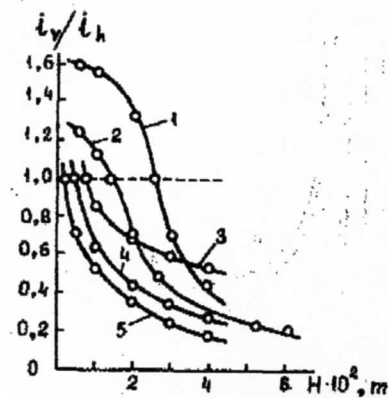


Figure 3. Changes of relations between schlieren ice segregation depth of the carbonate sapropel sample due to the temperature - humidity freezing conditions:

- 1 -  $t_0=-9.9^\circ\text{C}$ ,  $W_0=110\%$ ,  $\gamma_f=0.60 \text{ g/cm}^3$ ;
- 2 -  $t_0=-5.1^\circ\text{C}$ ,  $W_0=122\%$ ,  $\gamma_f=0.56 \text{ g/cm}^3$ ;
- 3 -  $t_0=-9.9^\circ\text{C}$ ,  $W_0=183\%$ ,  $\gamma_f=0.42 \text{ g/cm}^3$ ;
- 4 -  $t_0=-5.1^\circ\text{C}$ ,  $W_0=183\%$ ,  $\gamma_f=0.42 \text{ g/cm}^3$ ;
- 5 -  $t_0=-2.1^\circ\text{C}$ ,  $W_0=183\%$ ,  $\gamma_f=0.42 \text{ g/cm}^3$ .

increasing of initial water content of organogenic soil. The predominance of the contribution of the horizontal schlieren formation to the process of structure formation begins at lower  $grad t$  values for the less humid sample than for the sample with high  $W_0$  and the transfer point moves inside the frozen zone (Fig.3).

The cryogenic microstructure of sapropel shows regular and monotonous transformation with the changing of temperature conditions of freezing. As the temperature gradient lowers so does the freezing rate, the microstructure of high-ash containing sapropel changes from nearly massive with few subvertical layers to incompletely lattice-like and lattice-like (at domination of the vertical component), then at further lowering of the temperature gradient to the lattice-like with prevalence of the horizontal lenses and schlieren, horizontal-layered, and at significant slowing down of the freezing front to ataxitic with a sharp predominance of ice component. Dimensions of ice schlieren and organomineral blocks tend to increase as the temperature gradient decreases in the frozen zone (Fig.4) connected with the extending of the time of migration ice accumulation in the frozen and dewatering thawed part of soil when the migration water flows, predominantly form macro- and micro-structures.

The main re-structuring of the organomineral matrix of sapropel occurs within the limits of the frozen and freezing zones depending on the intensity of the local water migration processes, dehydration of the matrix and coagulation of the organic component. Increase of the freezing rate and temperature gradient in the frozen zone causes significant subsidence gradients of the frozen organogenic rock and intensive development of local dewatering process which results in the formation of a more compact microtexture.

The texture formation processes in sapropels of different types occur according to the general scheme while at the same time having significant differences. The research of freezing of carbonate sapropel ( $A^c=84\%$ ) has shown that the texture formation process begins in the thawed and continues in the freezing and frozen zones.



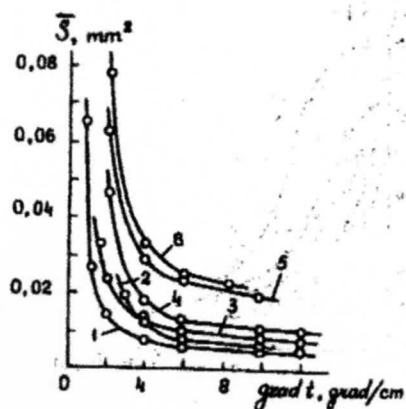


Figure 4. Correlation between the area organo-mineral layers and the temperature gradient in the frozen zone of various types of sapropel: Organic sapropel: 1-  $W_0=2000\%$ ,  $\gamma_f=0.048$  g/cm<sup>3</sup>; 2-  $W_0=710\%$ ,  $\gamma_f=0.13$  g/cm<sup>3</sup>; Siliceous sapropel: 3-  $W_0=379\%$ ,  $\gamma_f=0.24$  g/cm<sup>3</sup>; Mixed sapropel: 4-  $W_0=340\%$ ,  $\gamma_f=0.27$  g/cm<sup>3</sup>; 5-  $W_0=225\%$ ,  $\gamma_f=0.38$  g/cm<sup>3</sup>; Carbonate sapropel: 6-  $W_0=183\%$ ,  $\gamma_f=0.42$  g/cm<sup>3</sup>.

The most significant transformations of texture in the thawed zone occurs at low rate of freezing. Due to dewatering the particles of the organomineral matrix become nearer, the porosity decreases, regular texture starts to form with larger aggregates and blocks. A fast rate of freezing consolidation of the matrix goes on within the freezing and frozen zones. The forming ice framework deforms aggregates on the organomineral matrix caused by ice crystal growth which caused their local dehydration with driving part of the weakly combined water into the interstices and its subsequent freezing. Local dewatering is accompanied by aggregation and coagulation of the organo-mineral phase and formation of consolidated humus substances, water-resistant aggregates due to intensive cementing action.

Research of the microtexture of organomineral matrix of carbonate sapropel has shown its rather insignificant transformation in the changing of the temperature of freezing. So, at grad t values in the frozen zone equal to 10.2 grad/cm and 1.7 grad/cm respectively in the roof and floor or the sample and significant difference of microstructure, the texture of the organomineral matrix remains unchanged and is characterized by a uniform pattern of distribution of aggregates with sizes 5 to 15 microns with predominantly isometric shapes. Parts of weakly aggregated material 2-3 microns in size were also seen. Pores are not larger than 10 microns and their distribution pattern is uniform. Ice in cement is of a porous type.

The scheme of physico-chemical and texture formation processes in siliceous and mixed sapropel is qualitatively similar to their character in the carbonate sapropel. However, the higher humus content and, as a result, high natural humidity increases the significance of coagulation effects during the process of texture formation.

Cryogenic microstructure of the siliceous sapropel ( $AC=75.8\%$ ) is characterized by two main specific features. Formation of soil microstructure by means of differentiation of the system into ice and organomineral constituents depends to a significant extent on the freezing condi-

tions. At the same time the microtexture of the soil component varies rather inconsiderably within the given range of temperature - humidity conditions and is determined by the processes of local migration of water within the frozen zone. Microtexture of organomineral layers is characterized by uniform distribution of non-oriented aggregates 5 to 10 microns in size covered with a film of coagulation formations. The interstitial space is filled with fine-dispersed weakly oriented organomineral material with the size of 1-2 microns with tabular and flaky shapes. Ice in cement is presented by the porous type. Pore dimensions vary from 3 to 10 microns.

High water content characteristics of the mixed sapropel ( $AC=57.0$ ) in natural conditions leads to the formation of a cryogenic structure of an ataxitic type in it with the characteristic uniform distribution of organomineral grains at grad t > 4 grad/cm (Fig. 4). Microtexture of the organomineral grains is characterized by coagulation forms in the form of films surrounding quartz grains and calcite crystals, the presence of large grains (10-15 microns) cemented by consolidated and a dewatered fine-dispersed fraction. Interstitial pores filled with ice cement do not exceed 5 to 15 microns.

In low-ash organic sapropel ( $AC=12.7\%$ ) the formation of the texture of organomineral matrix is totally dependent on the coagulation effects. Consolidation of the matrix due to crystallization pressure causes dewatering of organomineral blocks, the occurrence of single flaky and filmy textural grains oriented along the surface of voluminous deformation of the block during local dewatering forming concentric casings surrounding mineral grains or ice crystals. The highest changes of the dimensions of structural grains occur at grad t values below 2.5 grad/cm at a slow rate of freezing (Fig. 4). Ataxitic cryogenic structure is characteristic of the occurrence of wide ice fields with "floating" organomineral layers which change orientation from subvertical to subhorizontal as the grad t values decrease.

Research of re-distribution of water content as a result of freezing of sapropels has shown that the humidity of the organomineral constituent can be regarded as near to the constant irrespective of the freezing conditions (Fig. 5). That is why the sharp increase of ice content due to intensive schlieren formation at freezing with low temperature gradients which significantly reduces the content of matrix-mineral component in the frozen sapropel is the main factor which decreases the efficiency of dewatering. Analysis of changes of organomineral constituent to the depth of the frozen zone has shown that after reaching certain (critical) values of temperature gradient grad t the complete elimination of organomineral layers and formation of a solid ice field occur. The grad t value is determined by the initial humidity of the sapropel and has higher values with an increase of  $W_0$ . For organic sapropel samples at initial humidity  $W_0=2000\%$ ;  $W_0=836\%$ ,  $W_0=710\%$ , grad t values are equal to 1.13; 0.82 and 0.73 grad/cm, respectively. For mixed sapropel at  $W_0=340\%$ , grad t = 0.46 grad/cm, for siliceous sapropel at  $W_0=379\%$  and 315%, grad t = 0.55 and 0.41 grad/cm.

The obtained values of grad t allow the determination of the optimal temperature and humidity conditions of freezing of organogenic soils at which the freezing mainly influences the matrix-mineral component of soil until the formation of ice horizon, as well as for

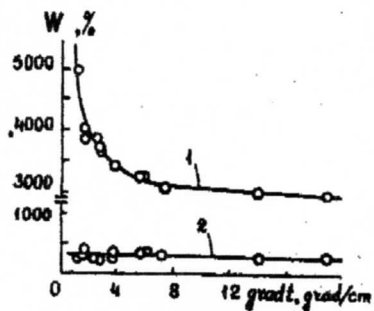


Figure 5. Correlation between the total humidity (1), the organomineral layers humidity (2) and the temperature gradient in the organic sapropel frozen zone  $W_0=2000\%$ ,  $\gamma_f=0.048 \text{ g/cm}^3$ ,  $A^C=12.7\%$ .

calculating the optimal depth of freezing for industrial purposes.

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