

## THE CONSTRUCTION PECULIARITIES OF LAYERED ORGANIC-INORGANIC SUPRASTRUCTURAL ANSAMBLE

S.Sh. GAKHRAMANOV

SPS "Selen" of National Academy of AR,  
F. Agayev, 14, Baku, Azerbaijan

The modern positions on the directed designing of supramolecular systems have been considered on the bases:

- Layered misfit compounds,
- clathrates and silicates with frame structures,
- layered hydroxides,
- layered structures with variable size of structural planes,
- layered compounds with variable size of structural cavities with "guests" of different size in "master" lattice,
- matrix crystals.

It is shown that the sizes and forms of matrix molecules and "guest" (cluster) molecules play the important role in the organization of the regularities of suprastructure layered misfit compounds, clathrate compounds and silicates with frame structures, different intercalated layered compounds and hydroxides.

It is established, that nonvalency chalcogen-chalcogen interactions play the special role in the creation of the supramolecular chalcogenide clusters.

### Introduction

The supramolecular physico-chemistry is the scientific direction in the center of attraction of which are the creation processes from the separate molecules of complex systems, connected in a single whole by the means of the intermolecular interactions. These interactions keep the fragments together – the electrostatic forces, hydrogen connection, Van der Waals forces in a whole are significantly weaker, than covalent connections in the molecule itself. For the increase of the durability of connections, the big enough joining "blocks" are used in them. The developed system of the connections because of the big surface of the contact is created. The ability to the molecular recognition is the main property of self-organizing supramolecular objects. The component recognition supposes the "complementary", - the mutual accordance of ensemble participants (substrate and receptor) as the geometric, so on the level of the creation of intermolecular connections [1]. The author of the given paper [1] pointed the different scientific disciplines as the objects of supramolecular chemistry, especially emphasized the set of the objects of inorganic and organic chemistry.

In the paper [1] it is emphasized, that intercalates, clathrates and structures of the type "master-guest" exist only in solid state. These structures aren't discrete supramolecules, and are referred to the solid supramolecular ensembles. The ensembles of the "guests" in "longitudinal" structures of the "master" and discrete supramolecules are emphasized. The "longitudinal" structures are the one-, two- or three-dimensional continuous or the half-continuous crystal creations with ion or covalent connection character. Nowadays, the circle of these objects (and common definitions and conceptions to them) is drawn approximately. However, the some classification of objects of supramolecular inorganic chemistry is present. The intercalates with "guests" of different size in "master" degree, clathrates, silicates with frame structures, complex phosphates of molybdenum and vanadium are referred to them [2]. Here [2] the discussion of the objects of inorganic supramolecular chemistry is limited by the compounds by the type "guest-master". They give the possibility of the studying

of the ensembles, in which the "guest" molecules create the strong connections with longitudinal structures. These structures influence on the geometrical and typological correspondence of the components (complementary), and also on the peculiarities of their self-organizing. The studying of the given objects and also multiphase and other complex layered objects can give the information about the new mechanisms of their creation.

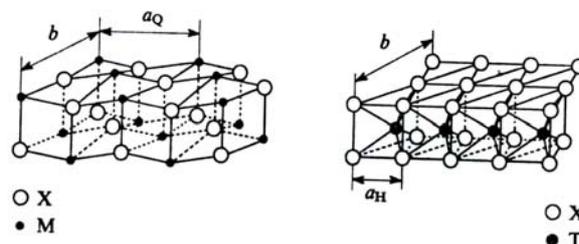


Fig. 1. The substructures MX (Q-layer) and TX<sub>2</sub> (H-layer) with disproportionate lattice parameters ( $a_Q$  and  $a_H$ ) [6].

The principle of geometric and typological complementary can be broken at the creation of the inorganic supramolecular ensembles. In the paper [2] the (as the systems "guest-master") layered intercalates (for example, in layered dichalcogenide matrix, graphite, clays) are especially emphasized. In them the layers in "master" lattice are connected because of Van der Waals interactions; the layers are shifted at the "guest" coming, and the change of interlayer distance is the sign of the intercalate creation [3]. The value of this change depends on the "guest" sizes and can be in 3-5 times greater, than the initial crystallographical parameter of "master" lattice. However, the limit intercalate stoichiometry is caused not only by the ration of "guest" sizes and holes in "master" lattice. This intercalate stoichiometry  $AaMX_2$  (A-metal,  $MX_2$  layered dichalcogenide matrix) is defined by the conditions of the filling of conduction band by the A donor electrons (i.e. by electron stabilization of whole  $AaMX_2$  structure), that is shown in papers [4-5]. The geometrical facts go on the second plane – it is proved by small sizes of M atoms in  $AaMX_2$  systems, the absence of the changes of

interlayer distances, small limit value  $a$  and dependence of the value  $a$  on valence  $A$  and  $M$  states (see fig.1 and 2).

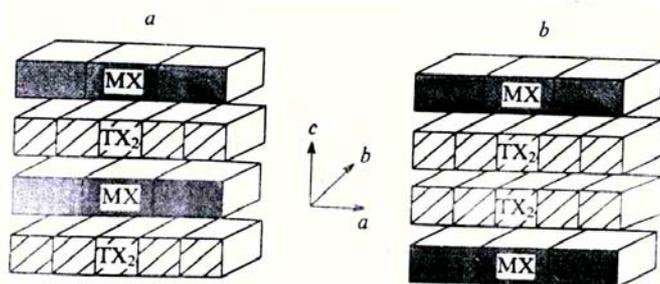


Fig.2. The scheme image of monolayer (a) and bilayer (δ) misfit structures [6].

So-called “matrix crystals” – supramolecular ensembles – synthetic opal with holes of the definite sizes, created the regular cubic sublattice are known. The quasi-lattice with enormously big parameters is formed at the filling of the holes (for example by the selenium – “guest”). The geometric disproportion of sublattices is compensated by the deformation of one from them (“guest”).

Another example of the disproportion of “guest” and “master is the variety of intercalates in layered dichalcogenide matrix – misfit compounds. They can be referred to the objects of supramolecular chemistry. These compounds present themselves the suprastructures, consisting from the structural elements, disproportionate in one crystallographical direction. It is need also to consider the peculiarities of structure self-organization of misfit layered compounds – synthetic chalcogenides with general formula  $(MX)_{1+x}(TX_2)_m$  ( $M-Sn, Pb, Bi, Sb, Ln; T-Ti, V, Nb, Ta, Cr; X-S, Se; x=0,08-0,28; m=1,2$ ) (fig.2).

The properties of misfit compounds should be defined significantly by the power competition, responsible for the saving of the hardness of disproportionate substructures (layered dichalcogenide matrix  $TX_2$ , from the one side and cubic structure of MK-c “guest” – from the another one, and strengths, connecting them in united suprastructure. At the same ratios of the given strengths the additive scheme of compound property estimation doesn't work. The disproportion leads to the defect creation, and also to the fragmentation of one of sublattices, influencing on compound properties [6].

The further consideration of the given problem is connected with nano-chemistry objects.

The nano-chemistry shows, that classic equations of chemistry kinetics aren't applicable at particle nano-sizes. The excess energy of surface atoms, the part of which is big in the relation to the inside atoms, significantly influences on the temperature of phase transfers in nano-particles. If their sizes are comparable for example with delocalization radius of charge carriers, the nonlinear electric phenomena appear. The appearance of new original methods of nano-particle creation is connected with this.

The region of nano-chemistry, which has the aim to create the closed or half-closed nano-and micro-reactors, is wide. For example, the polymers are created, in which the monodisperse empty spheres occupy 75% of volume. The diameter of these spheres can be regulated, beginning from the nano-sizes (10 nm), finishing by (1000 nm). The

molecular constructions are created (by type “vaz”), able to “catch” of “guest” molecules and release them. The molecular traps - karseplexes are synthesized. All these structures are related to the class of molecular containers, which are able to catch the given molecules and release them at the change of conditions [7-9] (fig.3).

The nano-chemistry creates the own objects – nano-particles, nano-reactors, nano-containers from the one side, and from the other one – uses the advantages of nano-particles, their special and flexibly variable properties for the own needs. Thus, two key conceptions are already defined – nano-particle and nano-reactor. The nano-particle characterizes the size parameter. The nano-reactor defines the nano-particle function. For example, *Fe* cluster almost totally loses its special properties and approximates to the metallic iron at the atom number in cluster  $n=15$ . At  $n>15$  it stays as the cluster in size meaning, but it loses qualities of “nano-reactor”, in which the qualities become the size function.

The nano-holes in different porous materials can be calculated by nano-reactors till the state, when their structure and properties of reacting system depend on the size of holes. When these dependences disappear, the nano-holes become only nano-sized, in which caught particles behave as they would be in unlimited volume [7]. However, very often the separation of conceptions nano-particle and nano-reactor is impossible. Nevertheless, these conceptions are needed for the recognition of two sides of sized effect – as the pure scaled space and as the physico-chemical phenomena, when properties “guest-atoms” depend on the size (very important sides in nano-chemistry).

And now few words about nano-reactors. The “empty” tube silicon fibers – one-dimensional nano-reactors have been created. The quazi-two-dimensional nano-reactors – hard graphite-like planes, connected by flexible-chain bridges from carbonic chains have been synthesized. The distances between graphite-like planes in such reactors can be varied [10]. The opposite and continuous pressing – the widening of interplane space is achieved because of the stimulating of conformational transfers in полиметиленовых chains – from rolled in nod of conformation (planes are maximally approached) till longitudinal chain (planes are maximally shifted).

The descriptions of principally new organization of chemical substance on the level, when at the creation of different materials from the components the leading meaning has not their reacting ability, and correspondence of sizes and forms of molecules – “guests” – to the frame holes, constructed by other molecules, - “masters” (or to hole of another, more big molecule – master) are paid attention. Such principle of compound creation allows us to join the coordinationally-saturated molecules, not including into chemical interaction with each other, in submolecules, and submolecular crystalline phases, thermodynamically more stable, than impurity of initial components [11]. The clarity in above mentioned phenomena is put by interactions, with the creation of izostructural cluster compounds. The main conceptions of clathrate chemistry gave the beginning the real searches of submolecular creations. The investigations of clathrate compounds [12-14] helped to understand, why at total absence of valence chemical connections between molecules of initial components they are able to join in thermodynamically stable phase.

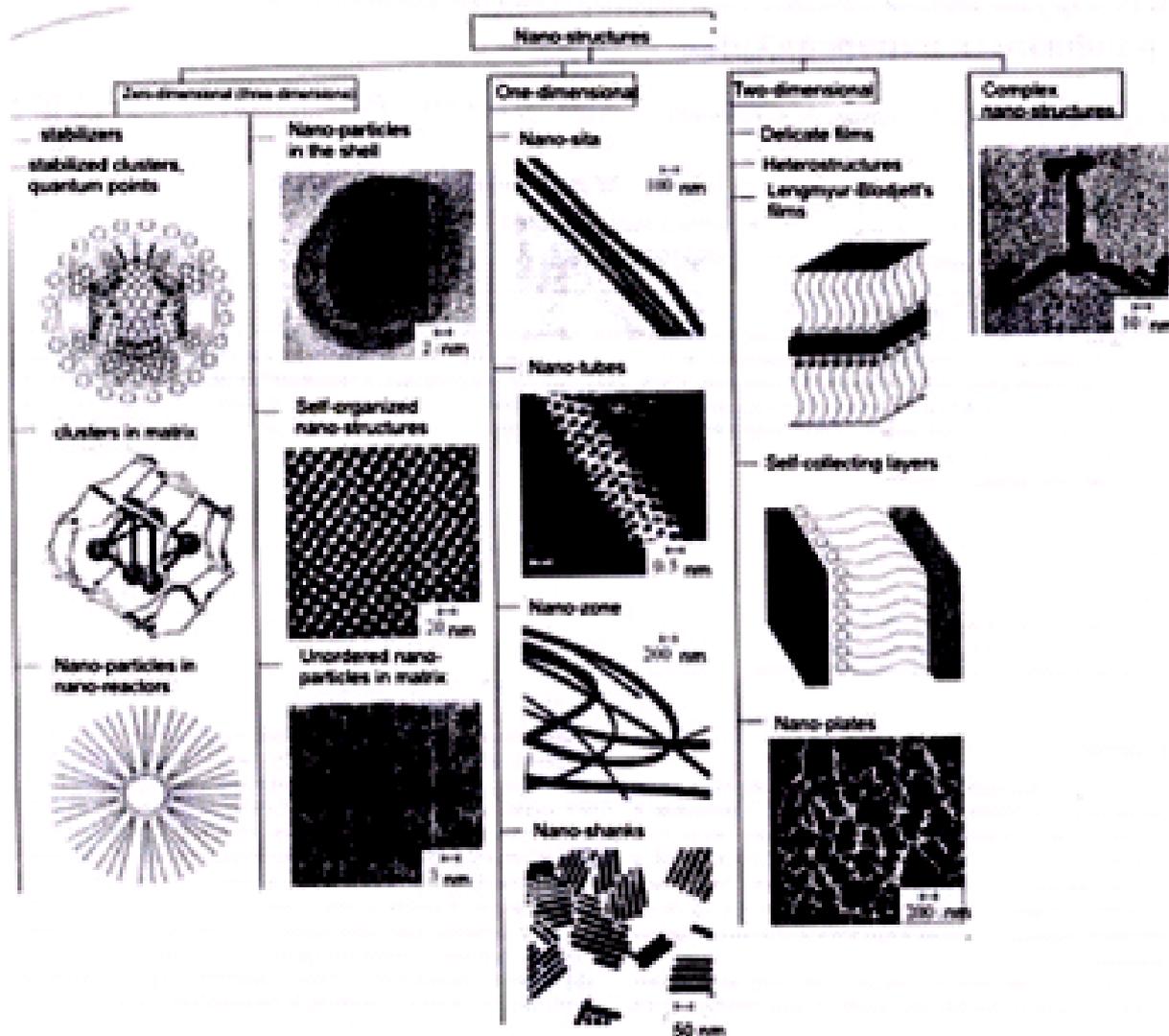


Fig.3. The nano-structure classification [19].

D.Len [1] emphasized the important role of invalency interactions in formation of similar compounds; the new conception "supramolecular chemistry", which he has defined as "chemistry outside the molecule", "chemistry of molecular ensembles and intermolecular connections" has been introduced by him.

The opening of clathrate-creating complexes of metals had been enriched the science of materials by many new objects. The revealing of supermolecular compounds allows to reveal one more thermodynamically profit mechanism of germ creation of new phase.

Thus, the necessity of the consideration of organization of chemical substance in the correspondence with sizes and forms of "guest" and "master" molecules of matrix has been became [15-19]. This is demonstrated on the fig.3.

The world of layered structures is rich; the methods of their creation and use are diverged. The community of layered systems is revealed in inorganic and organic world – in world of all nature systems. All this says about the unity of the mechanism of cleavage nature creation. Let's consider the one more example of the creation of layered structures.

#### Layered structures with variable size of structural holes

Such structures can be used in the capacity of two-dimensional nano-reactors. In these compounds the layers are connected between each other by Van der Waalse forces. Moreover, it is easy to change the size pf interlayer space. The layered structure increases the diffusion of diverge compounds in interlayer space and thus, eases the chemical modification of layered compounds.

In this relation the works, dedicated to the synthesis of nano-composites with the use of layered matrixes are interest. The compounds with negatively charged layers and cations in interlayer space – allumosilicates are widely used. However, such materials have the set of disadvantages, prohibited to the studying of formation mechanism of nano-structures in layered matrix.

Thus, the goal of the present paper is the review, analysis and revealing of common regularities in layered structures with elements "guests" in organic and inorganic suprastructures such as: clathrate compounds, eutectics,

misfit compounds and other layered systems with intercalates of different sizes in master lattice.

### Layered double hydroxides

The layered double hydroxides (LDH) of the composition  $M_{1-x}^{2+}M_x^{3+}(OH)_2[X_{x/n}^{n-} \cdot mH_2O]$  ( $X^-$  anion) are the more widely-spread two-dimensional nano-reactors. The compounds with  $M^{2+}=Mg^{2+}, Zn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Cd^{2+}, Sn^{2+}$  and  $M^{3+}=Al^{3+}, Fe^{3+}, Cr^{3+}, Mn^{3+}, Ga^{3+}, In^{3+}, Bi^{3+}$  are present. As a rule, the radiuses of cations  $M^{2+}$  and  $M^{3+}$ , participating in formation of layered structure, shouldn't differ more, than in 1,5 times. Practically any anion or anion complex can be in the capacity of the anion  $X^n$ . LDH structure presents itself the system of positively charged hydroxide layers  $[M_{1-xd}^{2+}M_x^{3+}(OH)_2]^x$  and anions, situating in interlayer space [20]. Structure of layered double hydroxides is presented on the fig.4.

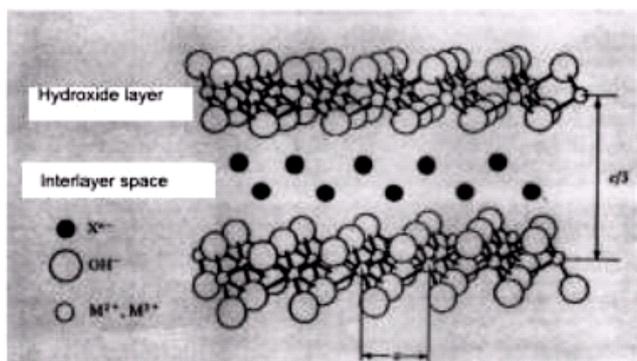


Fig.4. The scheme image of the structure of layered double hydroxides [7].

Besides anions in interlayer space the labile aqua molecules are present often. Given structure is stable because of the electrostatic interaction between positively charged hydroxide layers and interlayer anions, bringing the negative charge [20].

The anion situation in interlayer space can be diverged. As a rule, anions are situated in interlayer space thus, that in order to minimize its size.

LDH have the set of unique properties, important for the directed synthesis of nano-materials. The one of such properties is the stability of layered structure LDH in wide range of change of the sizes of cations and anions. Thus, samples LDH *Mg-Al* have been obtained, containing the different anions with sizes from 0,3 till 5 nm in interlayer space.

Thermal decomposition of LDH carries out with saving of layered structure that allows us to carry out the chemical reactions with the participation of anions of interlayer space at the high temperatures without the matrix decomposition [20]. Many functional properties of materials on the base of LDH connect with especial properties of intercalated anions in them.

The given properties open the wide possibilities for the chemical design of nano-composite materials on the base of layered double hydroxides.

The existence of hard hydroxide layers in LDH structure, limiting the interlayer space, creates the conditions for the synthesis of nano-systems, similar with conditions of their synthesis in nano-reactors. In [20] the composite obtaining, consisting from the nano-particles of *CdS* in interlayer space of LDH, is described. At thermolysis of *Cu*-consisting LDH at 400°C the creation of spherical particles of cuprum, by the size 2-5 nm (in interlayer space of matrix) and 5-8 nm (on surface) take place. At thermolysis of *Ni*-consisting LDH the spherical particles of *Ni* by diameter ~5 nm with very narrow particle distribution on sizes are formed. The increase of thermolysis temperature till 800°C leads to the increase of particle sizes till 15 nm with simultaneous distribution widening. And at thermolysis of *Co*-consisting LDH at 400-800°C the large (~100 nm) metal particles in form of discs, the size and morphology of which practically don't depend on thermolysis temperature, are created.

Thus, the use of nano-reactors opens the possibility for the design of functional nano-materials with given physicochemical parameters. Moreover, the solid-state matrix allows to avoid the aggregation of nanopartricles and defend them from external influences. The semiconductor layered materials by type: *GaSe, Bi2Te3, Sb2Te3, InSe* and others can be such solid-state matrixes. Thus, the given examples prove the advisability of use of layered structures in the capacity of two-dimensional nano-reactors.

### The consideration of design principles of supramolecular compounds with the use of organic cucurbituril

Such work was made by authors of papers [9,21-22] on organic macro-cycle cavidants – cucurbit[*n*]uril,  $[C_6H_6N_4O_2]_n$  ( $n=5-10$ ). The existence of hydrophobic intermolecular hole equally with polarized carbonyl groups of portals of cucurbiturils causes the high specificity of the creation of compounds by the type “guest-master”, is shown by them. The possibility of cucurbiturils to be in the capacity of the synthetic containers, in which the biomolecular reactions between specially chosen “guests” carry out with high regio- and stereoselectivity, has been considered [9].

The introduction of gas molecules, metal ions, organic molecules into hole leads to the creation of stable compounds, having the unusual structure and interest properties. In review of paper [9] the synthesis and structure of supramolecular materials, obtained with the use of macro-cycle cavidants, having the trivial name as cucurbiturils, are considered.

### The compounds of cucurbit[6]uril by type “guest-master”

The cucurbituril of  $C_{36}H_{36}N_{24}O_{12}$  composition (cucurbit[6]uril (5)), constructed from six glycoluril fragments, connected by methylene groups has been studied more well.

The existence of enough hard intramolecular hole causes the possibility of cucurbit[6]uril (5) the introduction of small guest molecules.

The creation of the compounds has been proved by crystallographically, and also by different physicochemical methods [9]. The cucurbit[6]uril (5) creates the stable compounds of the introduction with amines and diamines, alkyl and benzylammonium ions, dye molecules.

### The supramolecular compounds of cucurbit[6]uril with cluster aquacomplexes of metals

The use of volume fragments, not having its geometry, able to create the developed system of intermolecular connections because of the big contact surface, is the important condition of directed constructing of nano-sized complexes or supramolecular compounds. The cluster aquacomplexes of molybdenum and tungsten are suitable enough large molecular constructing blocks for the creation of supramolecular compounds with cucurbit[6]uril (5) [9].

The given compounds have chain structure, in which the supramolecules of structure type "barrel" with two coverings are connected between each other because of the short ( in the comparison with sum of Van der Waalse radiuses, which is equal to 3,9Å of nonvalency interactions Se.....Se (3,59-3,72Å). The hole cucurbit[6]uril in the compound  $\{[W_3Se_4(H_2O)_6Cl_3I_2(P_3HCC_{36}H_{36}N_{24}O_{12})]Cl_3 \cdot 18H_2O$  has the pyridine cation.

The interactions of chalcogen-chalcogen between neighbour clusters, which are character for three-nucleus of chalcogenide clusters of transfer metals [9].

Chalcogen-chalcogen interaction between neighbour cluster fragments  $M_3Y_4$  of supramolecular compounds is similar to interactions between chalcogen atom layers in layered dichalcogenide transfer metals  $MY_2(Y=S,Se)$  [23].

The comparison of the structures of chain supramolecular compounds

$\{[Wo_3Se_4(H_2O)_8Cl]_2(C_{36}H_{36}N_{24}O_{12})\}Cl_6 \cdot 16H_2O$  and  $\{[Wo_6HgSe_8(H_2O)_{14}Cl_4](C_{36}H_{36}N_{24}O_{12})\}Cl_4 \cdot 14H_2O$  [3] shows that they relate to each other as matrix and intercalate from the construction point of view: mercury atom is introduced between chalcogenide clusters  $M_3Y_4$  practically don't change the main parameters of supramolecules. Thus, as the authors of paper [9] conclude, the chemistry of supramolecular compounds of chalcogenide clusters is connected with chemistry of layered compounds more close, than it would be expected. In both cases the nonvalency interactions chalcogen-chalcogen play the important role in the structure creation.

### Conclusion

The considered review and analysis of cucurbiturils clathrates, layered dichalcogenides allow us to consider the organic and inorganic systems in the capacity of the molecular constructing fragments for the directed construction of nano-sized ordered layered supramolecular compounds. In hole of one frame the introduction of different "guests" and also clusters leads to the interaction between them and it becomes the motive force of creation of supramolecular compound. It is established, that in chemistry the nonvalency interaction chalcogenide-chalcogenide play important role in structure creation.

The peculiarities of synthesis of functional nano-composites on the base zero-, one- and two-dimensional solid-state nano-reactors, connected with formation of holes (zeolites, pores of mezoporous matrixes) or with interlayer holes of layered compounds. With the help of the intercalation method of dichalcogenides and metals, it is easy to create the layered crystals, using the longitudinal structural blocks  $TX_2$  and introduced reagents in solution at  $T=300^\circ K$ .

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**S.Ş. Qəhrəmanov**

### **LAYLI QEYRİ-ORQANİK VƏ ORQANİK SUPRASTRUKTUR ANSAMBLARININ QURULUŞUNUN XÜSUSİYYƏTLƏRİ**

Aşağıdakı supramolekulyar sistemlərinin istiqamətləndirilmiş quruluşunun müasir əsasnaməsi verilir:

-Laylı misfit birləşmələrin, klatratlarının karkas strukturlu silikatların, laylı hidrooksidlərin, laylı struktur ölçüsü dəyişən struktur müstəvilərinin, laylı birləşmələrin ölçüsü dəyişən struktur aralıqların müxtəlif ölçülü “qonaqlarının”, “qəbul edən qəfəsdə”, matrisli kristallar.

Göstərilir ki, suprastruktur laylı misfit birləşmələrinin, klatratların, karkas strukturlu silikatların, müxtəlif interkalyasiya olunmuş laylı birləşmələrinin, hidrooksidlərin qanunauyğun təşkil olunmasında əsas rol matris molekulların və “qonaqlar” molekullarının (klasterlərin) ölçüləri və formatları təşkil edir.

Təyin olunur ki, halkogenid supromolekulyar klasterlərin əmələ gəlməsində əsas rol halkogen-halkogen qeyri-valent əlaqələndirmələr oynayır.

**С.Ш. Кахраманов**

### **ОСОБЕННОСТИ КОНСТРУИРОВАНИЯ СЛОИСТЫХ ОРГАНО-НЕОРГАНИЧЕСКИХ СУПРАСТРУКТУРНЫХ АНСАМБЛЕЙ**

Рассмотрены современные положения по направленному конструированию супрамолекулярных систем на основе:

- слоистых мисфитных соединений,
- клатратов и силикатов с каркасными структурами,
- слоистых гидроксидов,
- слоистых структур с переменным размером структурных плоскостей,
- слоистых соединений с переменным размером структурных полостей с “гостями” различного размера в решетке “хозяина”,
- матричных кристаллов.

Показано, что в организации закономерностей супраструктурных слоистых мисфитных соединений, клатратов и силикатов с каркасными структурами, различных интеркалированных слоистых соединений, гидроксидов главную роль играют размеры и формы молекул матриц и молекул (кластеров) “гостей”.

Установлено, что в образовании супрамолекулярных халькогенидных кластеров особую роль играют невалентные взаимодействия халькоген-халькоген.

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