

A Brief Review on Formation of $(\text{H}_2\text{O})_n$ Clusters in Supramolecular Proton Transfer Compounds and Their Complexes

H. Aghabozorg^{a,*}, H. Eshtiagh-Hosseini^b, A.R. Salimi^b and M. Mirzaei^b

^aFaculty of Chemistry, Tarbiat Moallem University, Tehran, Iran

^bDepartment of Chemistry, School of Sciences, Ferdowsi University of Mashhad, Mashhad, Iran

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“Water clusters”, group of water molecules held together by hydrogen bonds, have been the subject of a number of intense experimental and theoretical investigations because of their importance in understanding cloud and ice formation, solution chemistry, and a large number of biochemical processes. “Water clusters” can play an important role in the stabilization of supramolecular systems both in solution and in the solid state and there is clearly a need for chemists a better understanding of how such aggregates influence the overall structure of their surroundings. The present brief review deals with $(\text{H}_2\text{O})_n$ clusters, identified/observed crystallographically upon 170 crystal structures of synthesized coordination compounds based upon proton transfer compounds by Hossein Aghabozorg’s research groups since 2000. The structural details of water clusters, $(\text{H}_2\text{O})_n$, $n = 2, 3, 4, 5, 6, 7, 8, 9, 15, 16$ and ∞ are described systematically.

Keywords: Supramolecular compounds, Proton transfer compounds, Coordination compounds, Water clusters, Hydrogen bonding, Water chemistry

INTRODUCTION

Water is of fundamental importance for human life and plays a critical role in many biological and chemical systems. It possesses polar hydrogen bonds (hereafter P-HB) which are responsible for a striking set of anomalous physical and chemical properties. Water molecules have two hydrogen atoms and two lone pairs enabling them to participate in four hydrogen bonds in a tetrahedral arrangement, but also frequently show 3-coordinate configurations. However, unlike covalent bonds, the P-HB geometry is much more flexible, Krygowski *et al.* describe the role of water molecules as a ‘gluing factor’ in organic crystals because of their readiness to deform from ideal P-HB geometry [1].

P-HBs can result in formation of diverse structures of water/water contacts directly as called water cluster, that is, $(\text{H}_2\text{O})_n$ clusters. “Water clusters” have extensively been studied both theoretically and experimentally. In fact, the presence of water is important in establishing H-bonded contributions to the total lattice energy, and is significant in establishing the stability of the hydrated crystal structure [2-4]. On the other hand, water could be viewed as playing a space-filling role in cases where there are cavities of a suitable size as a result of the close packing of the inorganic crystal structures. Moreover, the degree of structuring of a water cluster that can be imposed by its environment and vice versa can be of importance in designing new supramolecular coordination compounds containing proton transfer compounds.

Interest in the chemistry of such coordination compounds

*Corresponding author. E-mail: aghabozorg@saba.tmu.ac.ir

have recently increased. They are a broad class of compounds with considerable importance from both theoretical and practical points of view. The presence of water molecules can play an important role in stabilizing of these supramolecular species since the number of hydrogen bond acceptors and donors can differ significantly from those of the anhydrous compounds. Since, no comprehensive survey has been reported on solid-state coordination compounds, it seemed reasonable to begin with study of water/water contacts. The full picture is of course more complicated because most of the water molecules are also H-bonded equally strongly to donor/acceptor groups on the other molecules. Thus, we concentrate on the simpler case, with some comments on the total H-bond patterns, which often form extremely complex networks.

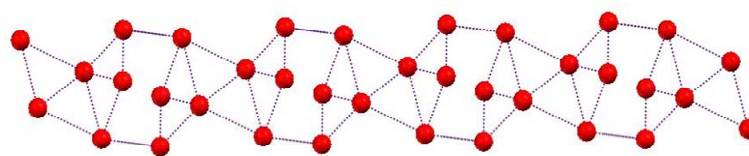
In this review, we have examined more than 170 hydrated supramolecular coordination structures retrieved from the reported structures by our research group. In our study, clearly we can categorize the cluster of water molecules on the basis of created shapes or the number of water molecules in any cluster. Herein, the presence of water clusters in the coordination compounds that have been synthesized by our research group, have been investigated with regard to inter- and intra-molecular interactions, especially hydrogen bonding interactions. Better understanding of hydrogen bonding interactions is dependent on knowing exact situation of any hydrogen atoms. On the other hand, it is difficult to determine the exact position of hydrogen atoms by single crystal X-ray technique. Regarding to this problem and for the refine of a hydrogen atom position theoretical method has been carried out. So, in order to obtain a certain definition in the study of the water cluster present in our synthesized compounds, we supposed here if the distance between two oxygen atoms in water molecules to be shorter than sum of their van der Waals radius that is equal 3.04 \AA , we may regard these interactions between water molecules to considering as a verified criterion in our investigation. Starting with good proton donors and acceptors would result in supramolecular proton transfer compounds, and then using a rich variety of metal ions including *s*-, *p*-, *d*- and sometimes *f*-block metals, supramolecular metal complexes, namely metal organic compounds could be built. Recently, Aghabozorg *et al.* in a brief review of about 140 structures has given the details about

the syntheses, crystal structures, spectroscopies, and solution studies of proton transfer ion pairs and their related metallic derivatives [5]. Accordingly, the remarkable significance of weak and strong interactions “beyond the molecule” including hydrogen bonds, van der Waals forces, ion pairing, and π - π interactions caused by different functional groups and their effects on explaining the structures is axiomatic. This brief review has mainly been performed upon our synthesized coordination compounds since 2000, which was reported in earlier review plus some other coordination compounds that are recently synthesized by our research groups. Approximately, a majority of them contain water clusters, which are in simple dimer-type water cluster shape. Indeed, twenty five crystal structures (%15) discussed among 170 crystal structures consisting of dimer-type water cluster. Herein, we have carried out a systematic study of water clusters, from a dimer to 9-mer, 15-mer, 16-mer, and ∞ that are present in our synthesized supramolecular coordination compounds with the general molecule formula of $(\text{H}_2\text{O})_n$. Regarding to the shapes of $(\text{H}_2\text{O})_n$ clusters, these observed clusters can exist in linear, branchy linear, simple cyclic, branchy cyclic, finite and infinite chains, and in the form of 2- and 3-dimensions that grow up in different directions.

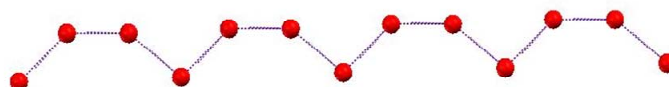
RESULTS AND DISCUSSION

The crystal structure of $(\text{pipzH}_2)_2(\text{btc}) \cdot 6\text{H}_2\text{O}$ (pipz = piperazine and btc = benzene-1,2,4,5-tetracarboxylic acid) [6] is containing two types of water molecule clusters that both of them shown by $(\text{H}_2\text{O})_\infty$ formula. In type A, the specific cyclic structure that connected together by corner developed in two dimensions; in type B simple infinite chain of water molecules without any branches. It is interesting to note that in this structure, there are two geometries of water molecule clusters. The numbers of water molecules with reduce order of water molecular cluster structures are in shape of the simple infinite chain, and it is similar to the pervious basic chain structures (Fig. 1). Crystal structure's of $(\text{pipzH}_2)(\text{pydcH})_2 \cdot 3\text{H}_2\text{O}$ compound (pydcH_2 = pyridine-2,6-dicarboxylic acid) [7] undertakes a $(\text{H}_2\text{O})_\infty$ cluster which is similar to the type B of previous compound and it is a simple infinite chain. However, unlikely, as can be seen in the Fig. 2, the two water molecule units alternatively repeat in a binary style while in the type B

A Brief Review on Formation of $(\text{H}_2\text{O})_n$ Clusters



Type A



Type B

Fig. 1

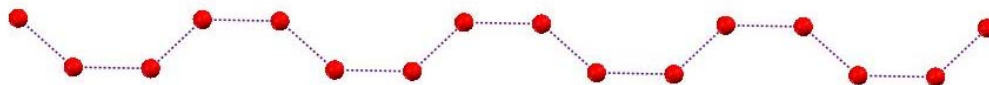


Fig. 2

cluster, water molecule units alternatively repeat in 1:2 ratio. Crystal structure of $(\text{pydaH})_2[\text{Cu}(\text{phendc})_2] \cdot 10\text{H}_2\text{O}$ compound ($\text{phendc} = 1,10\text{-phenanthroline-2,9-dicarboxylic acid}$ and $\text{pyda} = \text{pyridine-2,6-diamine}$) [8] is containing an interesting $(\text{H}_2\text{O})_\infty$ cluster which is shown in the Fig. 3. In this structure, huge three-dimensional network of water molecules have been created which has cavities with mineral and organic mixtures settled in. By devoting attention to the Fig. 3, the major ring of $(\text{H}_2\text{O})_\infty$ clusters which repeated in the three dimensional, reveals the huge water cluster that there was a quadruple ring of water molecules as a sub-ring. This cluster has been branched in two sites. In the direction of c -axis, an interesting view of this water cluster may reveal which it is like to a bird's wings that its wings could be convenient space for locating of other molecules inside of crystal structure. The $(\text{H}_2\text{O})_\infty$ cluster created by crystal structure of $(\text{pipzH}_2)_3[\text{Nd}(\text{pydc})_3] \cdot 15.33\text{H}_2\text{O}$ compound [9] forms an interesting feature in the c -axis, and these maintaining disordered water molecules (O11W, O7W and O9W) in the shape of branched star like structure (Fig. 4). The $(\text{H}_2\text{O})_\infty$ clusters present in the crystal structure of $(\text{pnH}_2)(\text{pydc})(\text{pydcH}_2) \cdot 2.5\text{H}_2\text{O}$ ($\text{pn} = \text{propane-1,3-diamine}$)

[10], forms an infinite branched chain-like water cluster which is branched in O2W and O4W positions. This chain almost alternatively is repeating in 1:2 ratio (Fig. 5).

Also $(\text{H}_2\text{O})_\infty$ cluster created by crystal structure of $(\text{pydaH})[\text{Cr}(\text{phendc})_2] \cdot 5\text{H}_2\text{O}$ [11], have formed a beautiful two-dimensional network of water molecules in which hexagonal rings repeats like benzene structure. An interesting view of this water cluster reminds us the crown ether structures (Fig. 6). The present water cluster in the crystalline network of $[\text{In}(\text{pydc})(\text{pydcH})(\text{H}_2\text{O})_2] \cdot 5\text{H}_2\text{O}$ [12] as $(\text{H}_2\text{O})_\infty$, is very similar to branched infinite chain of crystal structure in terms of water molecule cluster's shape [7]. Whereas the numbers of branches have been increased but the main body of the chain is still the same (Fig. 7). The $(\text{H}_2\text{O})_\infty$ clusters present in crystal structure of $(\text{pipzH}_2)_2[\text{Zn}(\text{pzdc})_2] \cdot 6\text{H}_2\text{O}$ ($\text{pzdc} = \text{pyrazine-2,3-dicarboxylic acid}$) [13] forms an infinite chain with 3:3 repeating ratio (Fig. 8). The $(\text{H}_2\text{O})_{16}$ cluster created by crystal structure of $[\text{In}_2\text{SO}_4(\text{pydc})_2(\text{phen})_2(\text{H}_2\text{O})_2] \cdot 5.5\text{H}_2\text{O}$ ($\text{phen} = 1,10\text{-phenanthroline}$) [14], forms a finite cluster which seems like a branched finite chain. The shape of this water cluster structure represents simple and single branches of

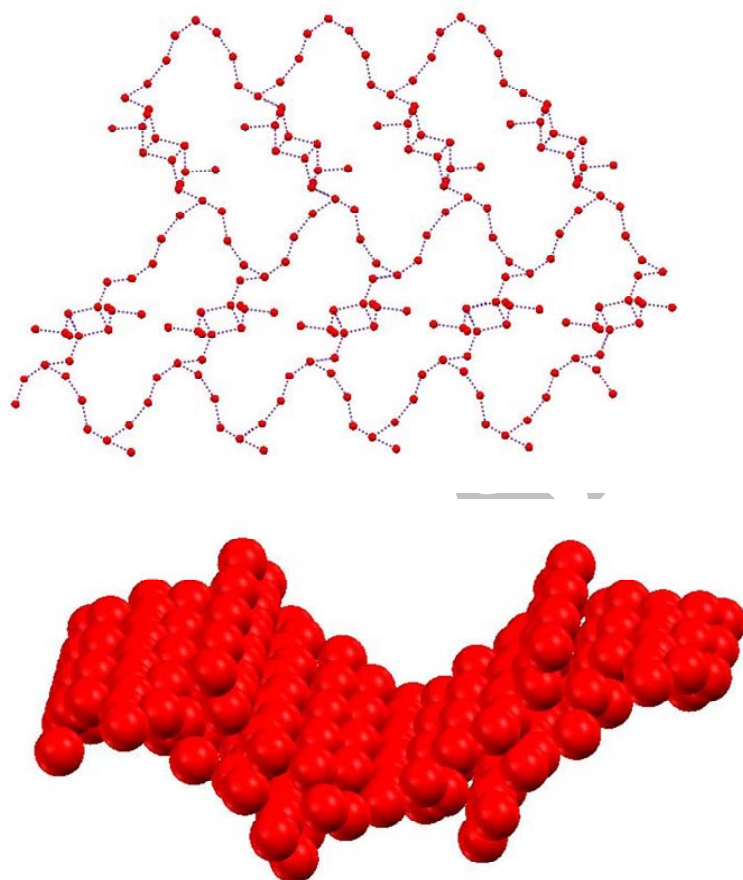


Fig. 3

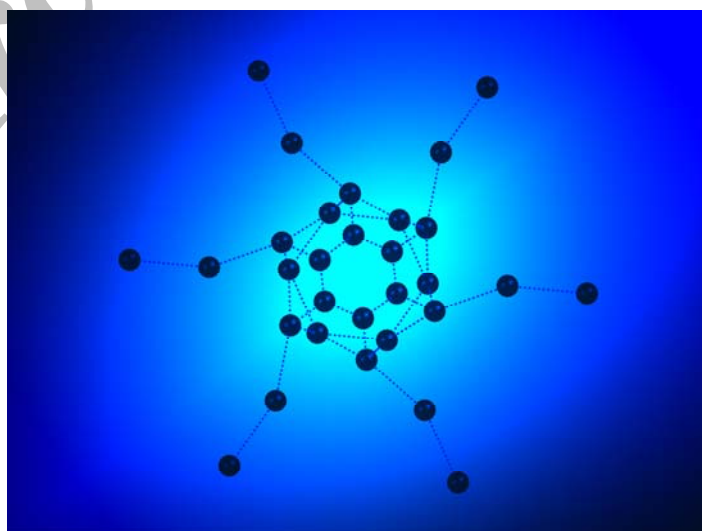


Fig. 4

A Brief Review on Formation of $(\text{H}_2\text{O})_n$ Clusters

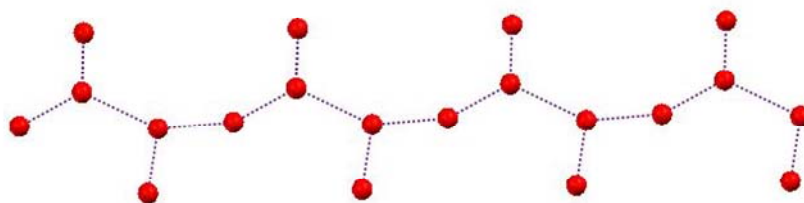
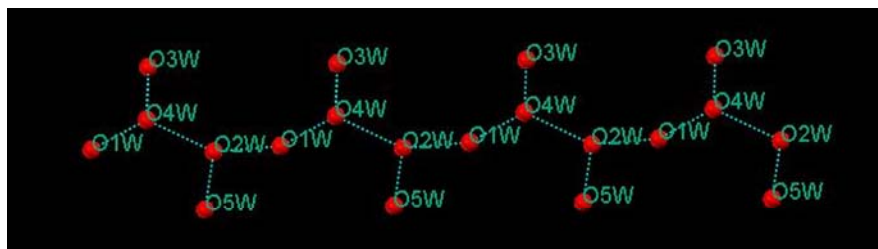


Fig. 5

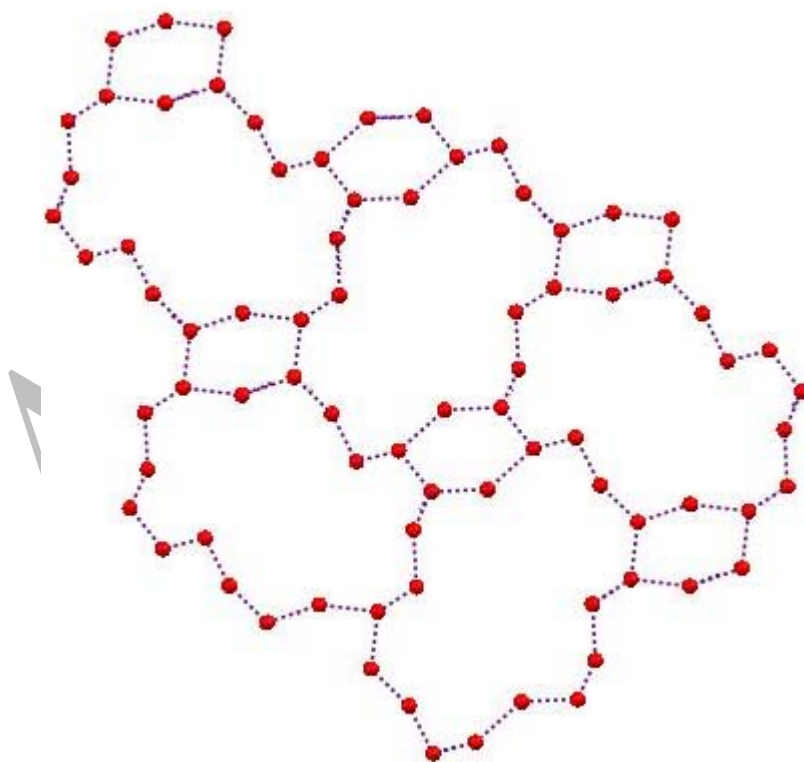


Fig. 6

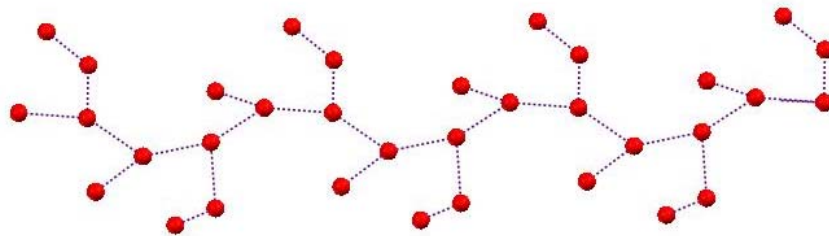


Fig. 7

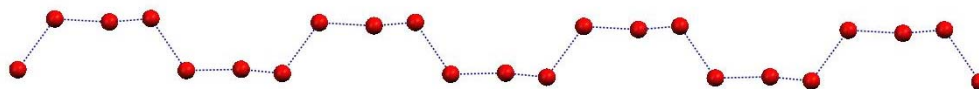


Fig. 8

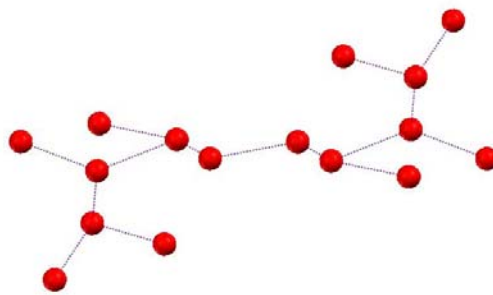


Fig. 9

water molecules on the main chain which includes 10 molecules of waters (Fig. 9). The $(\text{H}_2\text{O})_{15}$ cluster by crystal structure of $(\text{pipzH}_2)_3[\text{Nd}(\text{pydc})_3]_2 \cdot 15.33\text{H}_2\text{O}$ compound [9], as could make $(\text{H}_2\text{O})_\infty$ cluster in previous section, forms simultaneously a branched-cage cluster. The 10 water molecules form the cage cluster with 5 molecules as a branch in this cluster (Fig. 10). The $(\text{H}_2\text{O})_9$ cluster created by crystal structure of $[\text{Cu}(\text{hypdc})(\text{phen})(\text{H}_2\text{O})] \cdot 4.5\text{H}_2\text{O}$ ($\text{hypdcH}_2 = 4\text{-hydroxypyridine-2,6-dicarboxylic acid}$ and $\text{phen} = 1,10\text{-phenanthroline}$) [15], is placed after stated above-mentioned water clusters (Fig. 11). Crystal structure of $(4,4'\text{-bipyH})_2[\text{Sb}(\text{pydc})(\text{OH})_2(\mu\text{-OH})_2] \cdot 8\text{H}_2\text{O}$ ($\text{bipy} = 4,4'\text{-bipyridine}$) [16] contains $(\text{H}_2\text{O})_8$ cluster in the form of branchy cyclic. The ring of this water cluster is four-member and in the rhomboid form

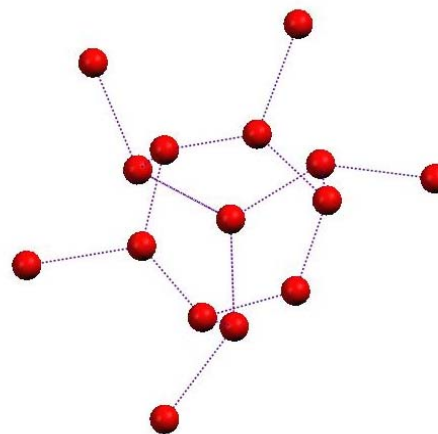


Fig. 10

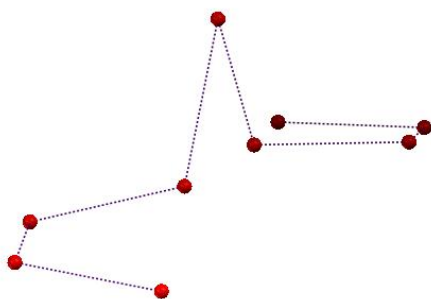


Fig. 11

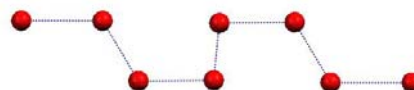


Fig. 14

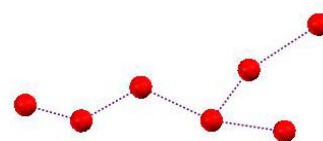


Fig. 15

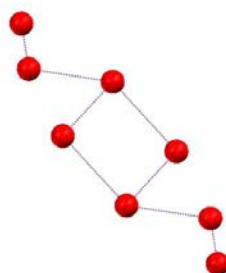


Fig. 12

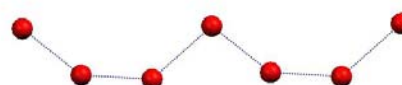


Fig. 16

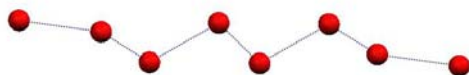


Fig. 13

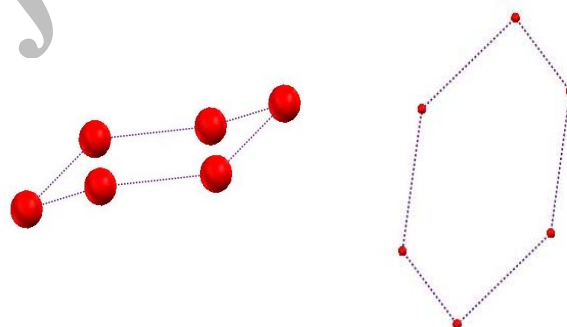


Fig. 17

that in the inverse corner has been located two branches as two molecules of water (Fig. 12). The $(\text{H}_2\text{O})_8$ cluster created by crystal structure of $(\text{pydcH})[\text{Ni}(\text{pydc})(\text{phen})(\text{H}_2\text{O})].4\text{H}_2\text{O}$ forms a simple chain as shown in Fig. 13 and also crystal structure of $[\text{Cd}_2(\text{hypydc})_2(\text{H}_2\text{O})_4].4\text{H}_2\text{O}$ contain a similar cluster that in this structure exits 2:2 repeat ratio which shown in Fig. 14 [17]. Crystal structures of $(\text{pnH}_2)(\text{pydcH})_2.2(\text{pydcH}_2).8\text{H}_2\text{O}$ [18] and $(\text{pipzH}_2)_2.(\text{btc}).6\text{H}_2\text{O}$ [6] ($\text{btc} = 1,2,4,5$ -benzenetetracarboxylic acid) are containing a one-branched chain of the water molecules and simple chain respectively, with the formula of $(\text{H}_2\text{O})_7$ (Figs. 15 and 16). In fact, the one-branched structure is a five-member as a basic chain that on one of the water molecules in the basic chain has been located two water molecules as branch form. Water

molecule cluster with the molecular formula of $(\text{H}_2\text{O})_6$ observed more than the other present form of water cluster structures in this review. Six crystal structures contain molecular clusters with $n = 6$ and they are structurally different than each other. Crystal structures of $(\text{bpyH})[\text{Al}(\text{pydc})_2].3\text{H}_2\text{O}$ ($\text{bpy} = 2$ -(2-pyridyl)pyridine) [19] and $[\text{Ni}(\text{ptcH})_2][\text{Ni}(\text{bpy})_4\text{H}_2\text{O}].6\text{H}_2\text{O}$ ($\text{ptcH}_3 =$ pyridine-2,4,6-tricarboxylic acid and $\text{bpy} = 2,2'$ -bipyridyl) [20] contain water clusters in the form of cyclohexane shape and with chair conformation which is shown in the Figs. 17 and 18, respectively. The molecule water cluster presents in $(\text{pipzH}_2)_3[\text{In}(\text{pydc})_3].12\text{H}_2\text{O}$ [21] is in the form of a simple chain which is similar to the structure reported in Ref. [12] (Fig. 19). The molecule cluster in the

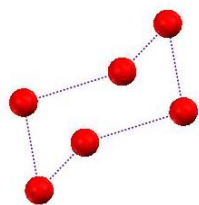


Fig. 18

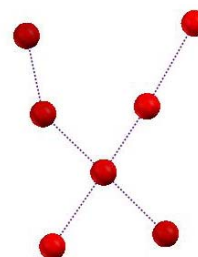


Fig. 22

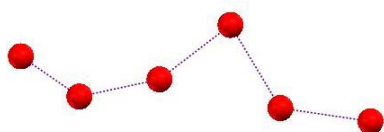


Fig. 19

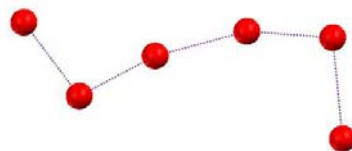


Fig. 20

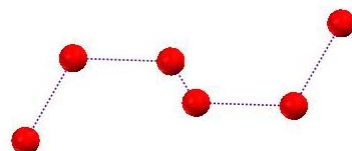


Fig. 21

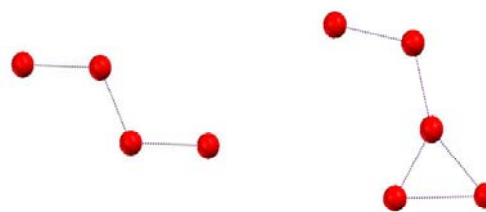


Fig. 23

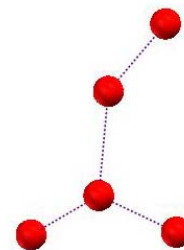


Fig. 24

$[\text{Ni}(\text{H}_2\text{O})_6][\text{Ni}(\text{pydc})_2] \cdot 3\text{H}_2\text{O}$ [22], shown in the Fig. 20, is in the form of a simple chain that is perpendicular to each other in the central part. The molecular cluster presents in the $(\text{tataH})_2[\text{Zn}(\text{pydc})_2] \cdot 10\text{H}_2\text{O}$ compound ($\text{tata} = 2,4,6\text{-triamino-1,3,5-triazine}$) [23], shown in Fig. 21, is also in the form of a simple chain (Related to $(\text{H}_2\text{O})_7$). The interesting structure of a molecular water cluster in the $(\text{UO}_2)_2(\mu_2\text{-OH})(\text{pdc})_2\text{Zn}(\text{bpy})(\text{Ac})_2(\text{H}_2\text{O})_9$ ($\text{pdcH}_2 = \text{pyridine-2,6-dicarboxylic acid}$, $\text{bpy} = 4,4'\text{-bipyridine}$, and $\text{HAc} = \text{acetic acid}$) [24] could be discussed in more details. In this structure in which a tetrahedral structure is appeared, four molecules of water gathered around a central water molecule and formed an aberrant tetrahedral and two water molecules are connected to it as a branch (Fig.

22). Similar to the crystal structure of $(\text{pipzH}_2)_2(\text{btc}) \cdot 6\text{H}_2\text{O}$ [6], crystal structure of $(\text{pnH}_2)[\text{Cu}(\text{pydc})_2] \cdot 4\text{H}_2\text{O}$ [25] have two types of molecular clusters with $n = 4$ and $n = 5$. Interestingly, one pentamer structure consists of three-member rings which have a branch from one vertex, but water clusters with $n = 4$ in the form of tetrahedral are as a simple chain (Fig. 23). The $(\text{H}_2\text{O})_5$ cluster in crystal structure of $(\text{phenH})_3[\text{Y}(\text{pydc})_3] \cdot \text{DMSO} \cdot 5\text{H}_2\text{O}$ ($\text{DMSO} = \text{dimethylsulfoxide}$) [26], possesses also a pentamer molecular cluster which is very similar to the previous structure. Whereas the base of 3 member rings has been opened because two water molecules get furthered and the rest of the structure is similar to the previous structure (Fig. 24). The molecular cluster, $(\text{H}_2\text{O})_5$, present in the

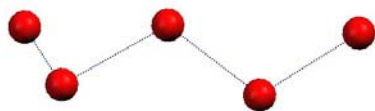


Fig. 25

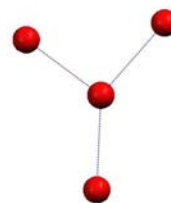


Fig. 27

(pnH₂)[Zn(hypydc)₂].3.5H₂O compound [27] is shown in Fig. 25 also is in the form of a simple chain. Within n = 4 water cluster structures, 2[AMPYH] · [ZnBr(Hmal)(mal)].2H₂O (mal = malonate, Hmal = hydrogen malonate, and AMPYH = 2-amino-4,6-dimethylpyrimidinium) [28], {[Sb(pydc)(phen)]₂O}.2DMSO·4H₂O [29], (pipzH₂)[Tl₂(pydc)₂Cl₄(H₂O)₂].4H₂O compounds [30] could further be the focus of attention. In these structures, water cluster is a four member ring. (pipzH₂)[Tl₂(pydc)₂Cl₄(H₂O)₂].4H₂O structure is as rhombus (Fig. 26a) whereas {[Sb(pydc)(phen)]₂O}.2DMSO.4H₂O structure is as square-planar (Fig. 26b), 2[AMPYH][ZnBr(Hmal)(mal)].2H₂O structure exists in a rectangular shape (Fig. 26c). The water molecular cluster with n = 4 in the crystal structure of [Cu(hypydc)(phen)].3H₂O [31] forms the triangle shape with three water molecules in which the last water molecule is in the center of a base chain (Fig. 27). The (H₂O)₄ clusters in crystal structures of [Sn(pydc)(phen)(OH)₂].3H₂O [26], (GnH)[Zn(pydc)(pydcH)].pydcH₂.4H₂O (Gn = guanidine) [32], (pipzH₂)[V(ox)₂O(H₂O)].2H₂O (ox = oxalic acid [33], (pipzH₂)[Al(H₂O)₆](SO₄)₄.8H₂O [34], (pipzH₂)[Cu(pydc)₂].4H₂O [35], (pnH₂)[Co(pydc)₂].4H₂O [25], and {[Ce

(pydc)(pydcH)(H₂O)₂].4H₂O} _n compounds [12] found in the form of a simple chain (Fig. 28). The (H₂O)₃ cluster created by crystalline networks reported in references [36-45] coordination compounds except the one in reference [38]. All contain (H₂O)₃ cluster with a V-shaped structure (Fig. 29).

CONCLUSIONS

According to the obtained results from investigations of water molecules present in our synthesized coordination compounds including proton transfer compounds can be conclude as follow: In coordination compounds, we can find numerous water clusters than proton transfer compounds with diverse geometries. Among observed geometries in water clusters, in similar to the different geometries of coordination compounds in inorganic chemistry aspect, we could also find that probability of formation of water clusters with n = 3, 4, 6, and ∞ is larger than the other geometries. In the case of n = ∞, casual directions of water molecules in whole crystalline structure may lead to the special stabilization of related structure. In the case of n = 6, cyclohexan-like directions of

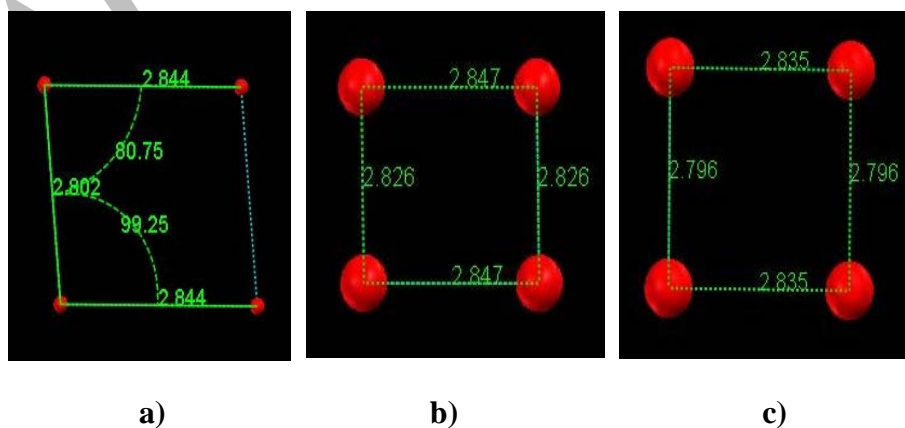


Fig. 26

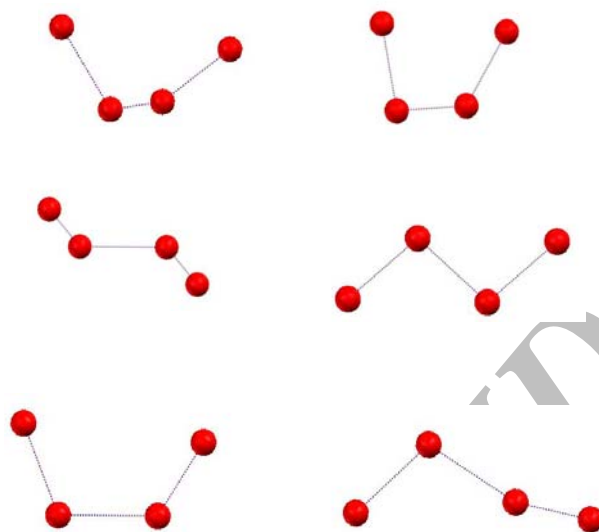


Fig. 28

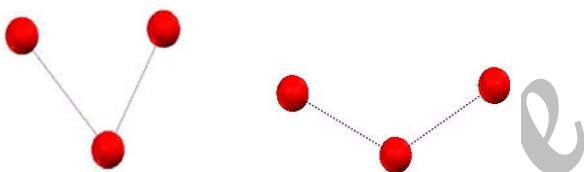


Fig. 29

water molecules have played an essential role in the formation and stabilization of final structure. In the cases of $n = 3$ and 4, one could find that V-form and simple-branched V-form geometries, respectively, were assigned as the probable forms for these categories clusters.

FUTURE PROSPECTS

Although searching other coordination compounds to find water clusters can be a good target point, but it should not be the whole final purpose. As is obvious, if a great fundamental research project cannot find its way to applied purposes, soon it will be forgotten. Thus, as a future prospect, finding the new geometries and significant range of water clusters formation in this category as well as new applications and practical usages of water clusters in medicine, pharmaceutical sciences, and the most logical reasons in their formation and creation will be an outstanding aim. However, from the viewpoint of constructing

functional proton transfer compounds, it will also become important to incorporate functional groups in those parts of them that act as ligands, instead of only using carbon and hydrogen containing moieties. In this way, the understanding and application principles of the "crystal engineering", as a main tool, will be helpful and effectiveness.

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