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Evidence for the existence of stable-water-clusters at room temperature and normal pressure

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ABSTRACT

We report the finding of isolated stable-water-clusters of tens of nanometers to micron size from the evaporation of very dilute sodium chloride solution at room temperature and normal atmospheric pressure. The stable-water-clusters are found to be electrically charged by examination via an Electric Force Microscope (EFM). Raman scattering and infrared spectrum of residues from the evaporation show similar but not identical characteristics of liquid water.

One of the greatest advances on our understanding of water comes from the study of water-clusters. Most water-cluster studies have been focused on smaller water-clusters, below 100 water molecules [1–5]. There exists a few studies on stable-water-clusters from tens of nanometers to micron size. These water-clusters are reported to be stable at room temperature at normal atmospheric pressures [6–11]. These clusters are reported to be created from very dilute solutions made from a variety of different materials.

Here we report further evidence of these stable-water-clusters created from diluting minute amounts of NaCl in ultra-pure water.

In order to reduce the possibility of contamination to our sample, we take the following steps:

1. Experiments are performed in a Class 100 clean-room (Industry standard) that requires room to be free of no more than 100 particles no larger than 0.5 microns per ft^3. Whenever it is necessary, we perform our experiment in an atmosphere-free glove-box or bottle, devoid of atmosphere and maintained a slightly positive pressure utilizing pure (99.999%) Argon gas so as to prohibit carbon dioxide contamination. Carbon dioxide can cause damage to the sample due to the fact that it dissolves easily in water and introduces unwanted ions in the water solution.
2. We use two commercially available systems that produce ultra-pure water which is characterized by its 18.2 MΩ cm resistivity. The large system, commercially available through Siemen's International, is used to provide bulk water for general use as well as feed water into the Purelab Ultra, Elga Lab Water, with TOC 3–10 ppb. This system is located inside the clean-room to protect against the introduction of contamination to our ultra-pure water during the preparation of samples and cleaning. We use a Laser Particle Counter (Lighthouse LS-60), which can detect and count separate particles ranging in size from 0.1 micron to 0.5 microns to ensure that our sample water has less than 10 particles no larger than 0.1 microns per 1 ml. Distilled water purchased from commercial supermarkets show particle counts of 5000 to 10,000 counts per 1 ml.
3. All utensils must be as clean as possible. We used the Lighthouse LS-60 Laser Particle Counter to ensure that all beakers are free of contamination after rinsing with 18.2 MΩ cm ultra-pure water by sampling the water for particles that might be left in the beakers after cleaning. It is critical to the success of this experiment that absolute care is taken to avoid contamination. Utensils, including containers, tubes, caps, pipettes, rotating flasks, condensing tubes, and slides that may be used to prepare the sample must be free from any contamination. We use only two kinds of containers: glass or those made from polypropylene. We shake/clean these utensils in ultra-pure water using ultrasound and then flush them with ultra-pure (18.2 MΩ cm) water. We repeat this process until we obtain a particle count of the flushed water to be below 100 particles or less, no larger than 0.1 microns per 1 ml.
4. We use NaCl with purity 99.99%.

In dilute solution of NaCl, sodium and chlorine ions coexist with water molecules, which have permanent electric dipole moment. The electric interactions of the dilute solution consist of those

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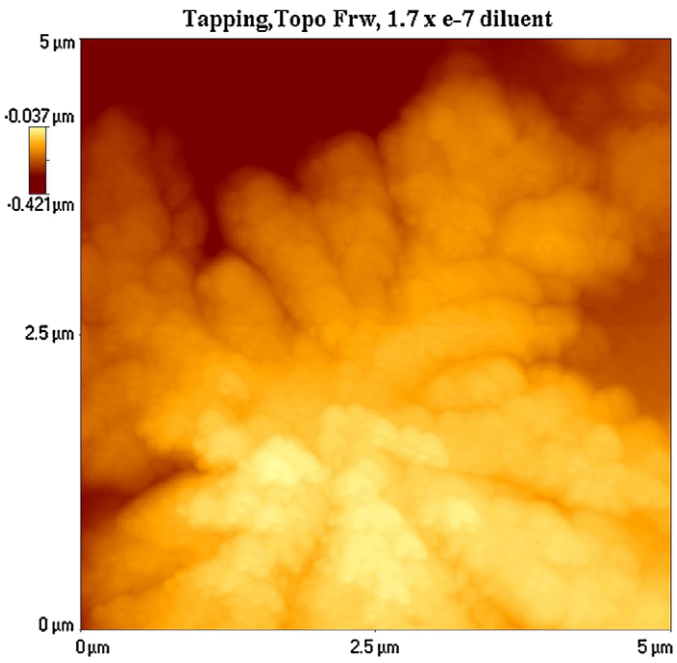

Fig. 1. Residues from evaporated 1.7×10^{-7} M NaCl solution taken with AFM and with EFM, shown in Fig. 2. For the AFM, the size of the picture is 5 µm × 5 µm. The different color code from bright to dark indicate the height of the clusters running from −0.037 µm to −0.421 µm. For the EFM picture (Fig. 2) the different color from bright to dark indicates the existence of electric potential from 1.257 V to −0.246 V. The different shades of color on the (Fig. 2) clusters indicate that there are charges on the surface of the clusters that generate electric potentials that are measured by the tip of the EFM.

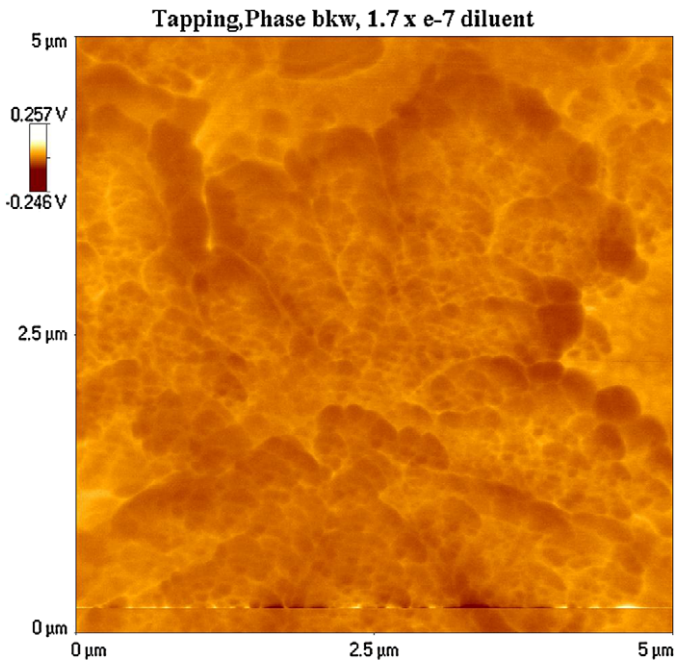

Fig. 2.

among ions and dipoles. When the density of ions is high, the dominant interactions are those among ions. As the concentration of NaCl decreases, there are less and less ions per unit volume. The interaction among ions becomes less and less. The interaction among dipoles becomes more and more important. There comes a point when the dipole–dipole interactions dominate. The transition point when this occurs is found experimentally to be at the concentration about 10^{-4} M(mol/liter) [6]. At concentration below this transition point, water molecules will attract to one another to form clusters that have permanent electric dipole moment, much like small magnets stick together to form a big magnet.

For this experiment we diluted our NaCl solution to 10^{-7} M, far below this transition point. We then place drops of the solution on a glass slide and wait for it to dry. The residue is visible under an ordinary light microscope. To detect more structures of clusters we use an Atomic Force Microscope (AFM), the Innova model made by Veeco with includes an EFM (electric force microscope) mode. The clusters are soft and tapping mode of AFM is necessary. In the forward motion of the tip in the tapping AFM mode, the topography of the clusters is shown. The electric force microscope (EFM), works simultaneously with the AFM. When the tip scans backward at a slightly higher height concurrent with an electric potential, it records and shows as a different shade of color representing the influence created by the different values of electric potential emitting from the sample. When there is no charge on the cluster, there is no change in electric potential and the color is unchanged. Any variation of color indicates that local charges are present on the sample. In Figs. 1 and 2 we show one set of AFM and EFM pictures taken of our sample.

As a control, we put drops of pure water on similar glass slide under similar conditions and let them dry — we see no such structures as in Fig. 1 in AFM pictures.

There is a great variety of snow-flake like shapes. Similarly, there is a great variety of stable-water-clusters. We report one specific type, which we believe may give more weight to the idea that these stable-water-clusters may have a permanent electric dipole. Since one isolate water molecule has permanent electric dipole moment, it is likely that the stable-water-clusters made up of numerous water molecules would have a permanent electric dipole moment.

Let us have a group of small magnets with permanent magnetic dipole moments. If we put many of them in a small space, they will clamp together. If we spread them out evenly, these magnets will line-up because of their contained magnetic forces. Instead of small magnets, we suggest stable-water-clusters with permanent electric dipole moments. When the density of clusters is high from evaporating 10^{-7} M on a glass slide, they will clump together as in Figs. 1 and 2. If the density of clusters is not as dense, the clusters will spread out in straight lines as shown in Fig. 3(a). Here, they form straight lines most likely due to the electric attraction between the permanent electric dipole moment of the stable-water-clusters.

There is an interesting point of the lines formed by the clusters in Fig. 3(a). All the horizontal lines intersect with the vertical line at 102°, which is the same angle sustained by two hydrogen atoms interacting with the oxygen atom in a water molecule. One might conclude that such could be accidental, but the authors suggest it is a manifestation of the scaling law.

Most of the pictures are not as regular as Fig. 3. In Fig. 4(a), we show another example, which has less regular structures. Such structures are also quite common in the distillate, which is obtained from using a standard glass distillation unit to distill the 10^{-7} M, NaCl. One drop of distillate is left to dry on a glass slide and the optical picture of its residue is shown in Fig. 4(b).

We consider the fact that the residue from distillate also contains similar structures as that of the original solution gives strong evidence that the clusters we see are made of water molecules and not from contaminants.

The AFM pictures from the residues of distillate, such as those shown in Fig. 4(b) are similar to those from the original 10^{-7} M NaCl solution, as shown in Fig. 1.

In order to ascertain the molecular content of these residues, we have obtained infrared spectra as well as spectra from Raman scattering:

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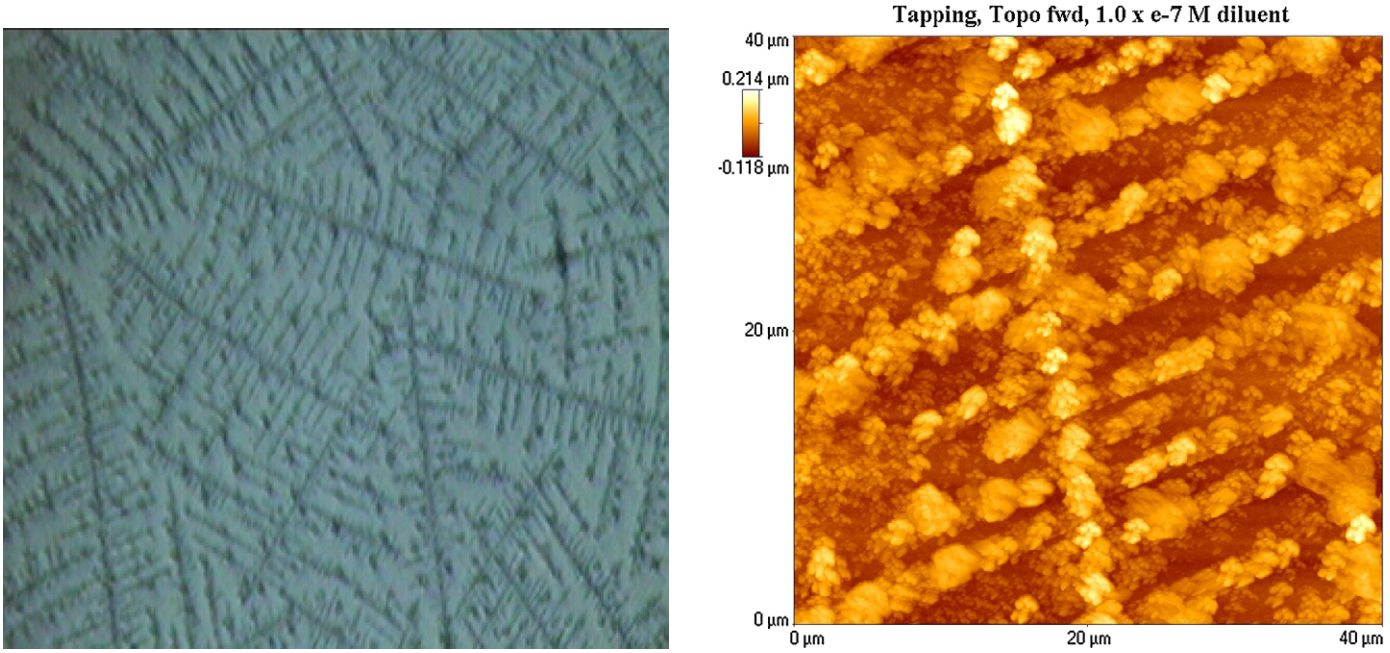
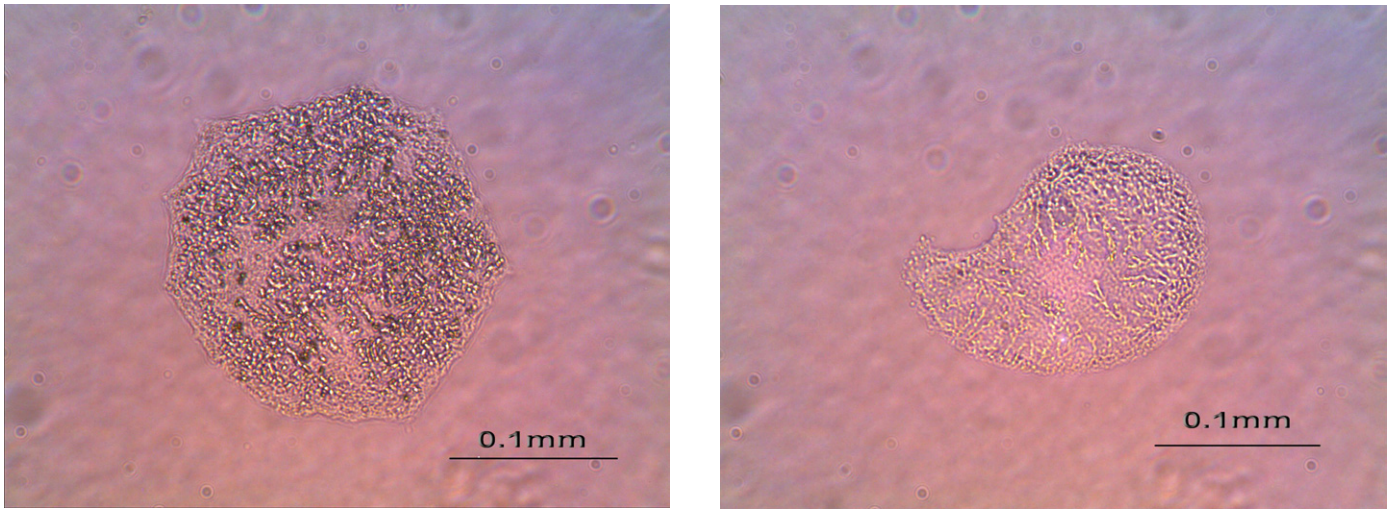


(a) (b)

Fig. 3. On the left (a), is a picture taken with an ordinary light microscope. On the right (b), is the AFM picture showing the straight lines shown in (a). The size of the AFM picture is 40 µm × 40 µm.



(a) (b)

Fig. 4. On the left (a) is the picture from an optical microscope of the residue of one drop of 10^{-7} M NaCl. On the right (b), is that of the residue of the distillate from 10^{-7} M NaCl.

(a) Infrared spectra:
To strengthen the signal of infrared spectra, we have obtained a large amount of solid residues by evaporating more than a hundred drops of 10^{-7} M NaCl on the same slide. For optimum results, one drop is allowed to dry before we add a second drop. Then a third drop, and so forth. The infrared spectrometer we use is FT-IR Nicolet 380 with ATR from Thermo Electron Corporation. The results are shown in Fig. 5.

(b) Spectra from Raman scattering:
We place one drop of 10^{-7} M NaCl on a silicon slide, which is coated with TiO_2. Then the residue is obtained on the surface of the silicon slide by placing it to dry in a dry box. We use a Raman microscope made by RENISHAW, UK, which uses an Argon ion laser with wave length of 514.5 nm and power 20 mW to sweep a range from 100 to 4000 cm^{-1}. The Raman spectrum is obtained from sweeping an area of 2 µm × 2 µm, where there is residue. The curves are shown in Fig. 6. We focus the Argon laser beam on two separate residue spots on the silicon slide, we obtain two curves SP2 and SP3. For comparison we also obtain the Raman spectrum of liquid water SP1 by focusing the Argon beam on to inside of a capillary which holds the liquid water. The dominant Raman peak of liquid water is at 3426.42 cm^{-1}. The Raman spectra of the residues do not have a peak there, but have three peaks next to it at 3145.33 cm^{-1}, 3049.51 cm^{-1}, 2860.34 cm^{-1}. There are, in addition, smaller peaks at 2011.24 cm^{-1}, 1754.81 cm^{-1}, 1709.31 cm^{-1}, and 1404.86 cm^{-1}, *which may be related to the Raman peak of liquid water at* 1651.52 cm^{-1}. It may be interpreted as the molecular structure of stable water clusters is quite similar but not identical to that of liquid water. For back-

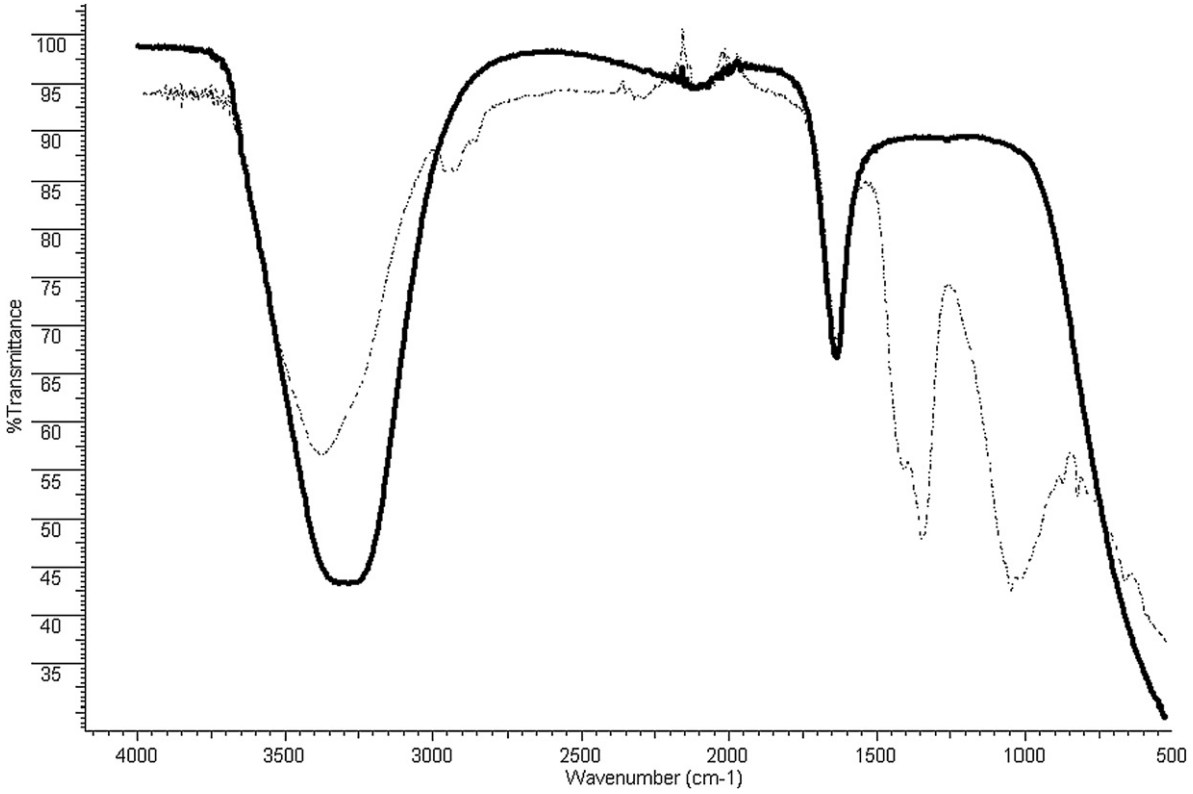


Fig. 5. Infrared spectra for pure water (solid line) and solid sample of stable-water-clusters (broken line). The absorption peaks for pure water are 3283.5 cm^{-1} and 1634.5 cm^{-1}. The absorption peaks for stable-water-clusters are 3371.4 cm^{-1}, 1639.5 cm^{-1}, 1342 cm^{-1}, 822.5 cm^{-1}.

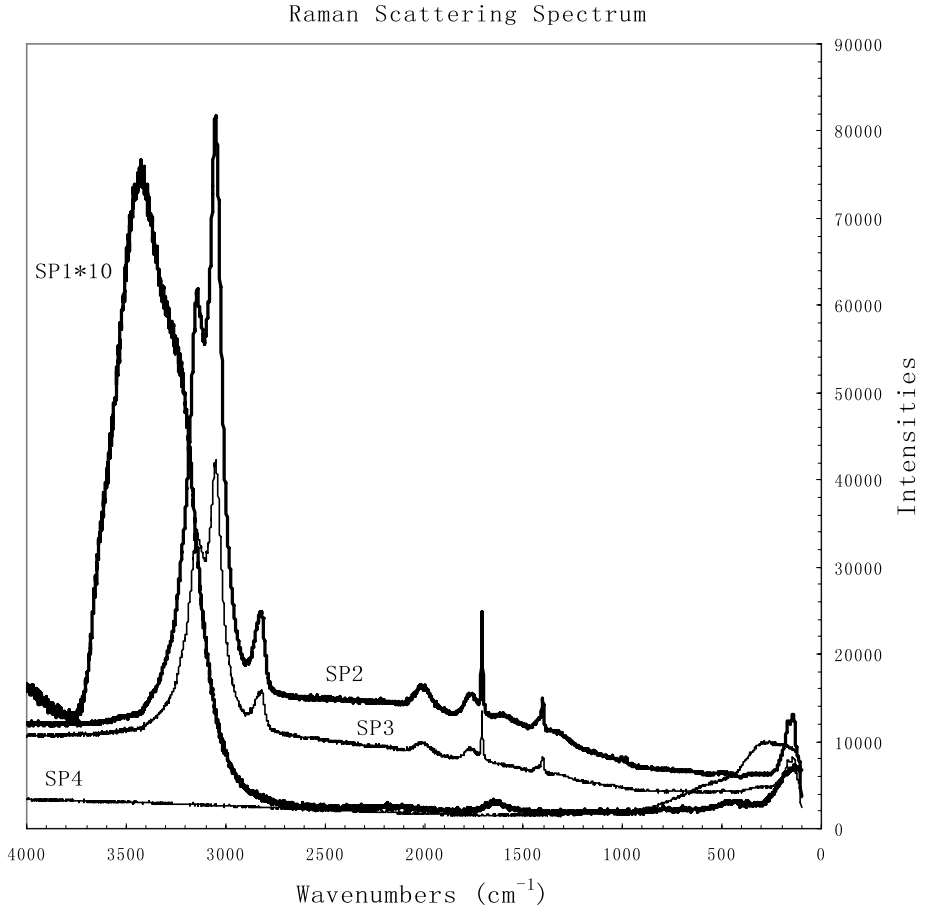


Fig. 6. Raman spectra: the Raman spectrum for liquid water is labeled as SP1, SP2, and SP3 represent the Raman spectra at two separate spots with residues on the silicon slide. SP4 is that of blank spot on the silicon slide where there is no residue.

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ground noise we also focus the Argon laser to spot on the silicon slide where there is no residue. We see a flat curve and no peak.

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