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ASTROCHEMISTRY

Molecular Adsorption on Cold Gas-Phase Hydrogen-Bonded Clusters of Chiral Molecules

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Abstract

Gas-phase molecular adsorption was investigated as a model for molecular cloud formation. Molecular adsorption on cold gas-phase hydrogen-bonded clusters containing protonated tryptophan (Trp) enantiomers and monosaccharides such as methyl-α-D-glucoside, D-ribose, and D-arabinose was detected using a tandem mass spectrometer equipped with an electrospray ionization source and cold ion trap. The adsorption sites on the surface of cold gas-phase hydrogen-bonded cluster ions were quantified using gas-phase N_2 adsorption-mass spectrometry. The gas-phase N_2 adsorption experiments indicated that the number of adsorption sites on the surface of the hydrogen-bonded heterochiral clusters containing L-Trp and D-monosaccharides exceeded the number of adsorption sites on the homochiral clusters containing D-Trp and D-monosaccharides. H_2O molecules were preferentially adsorbed on the heterochiral clusters, and larger water clusters were formed in the gas phase. Physical and chemical properties of cold gas-phase hydrogen-bonded clusters containing biological molecules were useful for investigating enantiomer selectivity and chemical evolution in interstellar molecular clouds.

Keywords Molecular cloud · Water cluster · Mass spectrometry · Ion trap · Electrospray ionization

Introduction

Biomolecules consist of L-amino acids and D-sugars (R-sugars), and water is known to play an important role in the structure and function of biomolecules. The origin of homochirality in biomolecules and water on Earth is one of the most important research issues in life sciences. Abiotic formation of amino acids, small peptides, and sugars has been investigated using simulated interstellar ice comprising mixtures of H_2O, CH_3OH, CO, CO_2, NH_3, and HCN (Bernstein et al. 2002; Muñoz Caro et al. 2002; Gontareva et al. 2009; Abplanalp et al. 2016; Furukawa et al. 2019), whereas the formation of biological molecules with enantiomeric excess has not been detected in the simulated interstellar ices.

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Circularly polarized light in star-forming regions could induce an enantiomeric excess of molecules in space (Bailey et al. 1998; McGuire et al. 2016). Enantiomeric excess of amino acids and sugars has been observed in meteorites (Cronin and Pizzarello 1997; Engel and Macko 1997; Cooper and Rios 2016). The current consensus is that water on Earth is derived primarily from a meteoric source (Robert 2001; Cleeves et al. 2014; Sarafian et al. 2014; O'Brien et al. 2018). The extraterrestrial origin of enantiomeric excess in biological molecules and water on Earth has been evaluated in numerous studies that are based on the fields of astrobiology and geophysics.

The preference for homochirality of amino acids in the gas phase and in solution has been investigated using mass spectrometry, infrared spectroscopy, and nuclear magnetic resonance spectroscopy (Nanita and Cooks 2006; Kong et al. 2006; Vandenbussche et al. 2006). Gas-phase serine octamers were stable and exhibited a preference for homochirality. Tandem mass spectral data of gas-phase cluster ions of serine and glucose indicated that D-glucose was more favorably incorporated into an L-serine octamer than its enantiomer (Takats et al. 2003). Gas-phase ion/molecule reactions in a cold ion trap have been analyzed in interstellar chemistry, given that the chemical reactions in space occur at low temperatures and low densities (Gerlich and Horning 1992; Gerlich and Smith 2006). We have investigated molecular recognition of tryptophan (Trp) in cold gas-phase hydrogen-bonded clusters, because Trp has been used as probes investigating structure and reaction of various proteins due to its high absorption intensity in the ultraviolet region (Callis and Liu 2004; Candace and Lee 2010). Enantiomer-selective photodissociation of cold gas-phase protonated Trp on a chiral crown ether was suppressed when the temperature exceeded 170 K, and no difference between the enantiomers was observed at 300 K (Fujihara et al. 2014; Fujihara et al. 2015). These temperatures correspond to those of interstellar and atmospheric molecular clouds, respectively. Enantiomer-selective reactions such as glycosidic bond cleavage and *C*-glycosylation were observed in mass spectrometric studies that elucidated the reactivity of cold gas-phase cluster ions containing Trp and sugars as a model for chemical evolution in interstellar molecular clouds (Doan and Fujihara 2018; Nguyen and Fujihara 2018).

Heterogeneous nucleation of ice on mineral dust, soot, organic molecules, and biological particles in the gas phase has been investigated extensively since the 1980s using continuous flow diffusion chambers, given that the heterogeneous nucleation is important for the formation of atmospheric molecular clouds (DeMott et al. 2011; Hoose and Möhler 2012). The cross section for attachment of water molecules to protonated water clusters was measured using a mass spectrometry-based technique (Zamith et al. 2010). The water adsorption propensity of globular conformations of protonated peptides in a drift tube was found to be higher than that of helical conformations (Kohtani and Jarrold 2004). The rate of water adduction in the gas phase differs for hexose isomers (Campbell et al. 2017). N_2 adsorption on small metal cluster cations depends on the structure of the cations (Patwari et al. 2011; Ohshimo et al. 2017). Hydroxy groups of carbohydrates have been quantified using gas-phase N_2 adsorption (Ochiai et al. 2020). The structures and conformations of gas-phase hydrogen-bonded cluster ions were found to affect the N_2 adsorption ability of the clusters (Fujihara and Shimada 2019).

In this study, the adsorption of N_2 and H_2O on cold gas-phase protonated clusters containing monosaccharide and Trp enantiomers (Fig. 1) is investigated as a model for molecular cloud formation. The monosaccharides used in this study, D-glucoside, D-ribose, and D-arabinose, contain four hydroxy groups. Based on these results, molecular cloud formation on chiral molecules and the implications of this process for chemical evolution have been discussed.

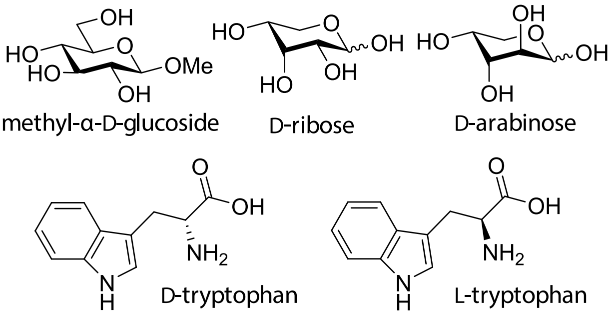

Fig. 1 Structures of chiral molecules used in this study

Methods

Molecular adsorption on cold gas-phase protonated clusters was observed using a tandem mass spectrometer equipped with an electrospray ionization source and a temperature-controlled ion trap (Fujihara et al. 2014; Fujihara and Shimada 2019). Protonated clusters of monosaccharide and Trp enantiomers were generated via electrospray ionization of solutions containing 0.5 mM of carbohydrate and Trp enantiomers in a mixture of water and methanol with 1% acetic acid. The cluster ions were transferred to the gas phase through a metal capillary and skimmer. The gas-phase ions were pulsed into a quadrupole mass filter and a temperature-controlled 22-pole ion trap using an octopole ion guide. The mass-selected ions were thermalized via multiple collisions with He buffer gas in the ion trap for 50 ms. The mass-selected and temperature-controlled ions in the gas phase were at low temperatures and low densities. N_2 adsorption measurements were performed with He buffer gas containing 20% N_2 at 50 K, and the N_2 molecules were adsorbed on the mass-selected ions in the ion trap. H_2O adsorption measurements were performed with He buffer gas at 8 K once the apparatus reached atmospheric pressure due to air leakage. The pressure in the ion trap was the same with that for the N_2 adsorption measurements, and the ambient pressure in the chamber containing the ion trap was approximately 2×10^{-4} Pa. H_2O molecules remaining in the ion trap were adsorbed on the mass-selected ions. The cluster ions with adsorbed N_2 and H_2O were extracted from the ion trap and subjected to mass-analysis using a reflectron time-of-flight mass spectrometer.

Results and Discussion

N_2 Adsorption on Heterochiral and Homochiral Clusters

Figure 2a illustrates the mass spectrum of H^+(L-Trp)(D-glucoside)$(N_2)_n$ formed via N_2 adsorption on the mass-selected hydrogen-bonded heterochiral H^+(L-Trp)(D-glucoside) cluster at 50 K. The term n denotes the number of N_2 molecules adsorbed on the mass-selected ions in the ion trap. The spectrum was normalized by setting the ion intensity for $n = 1$ to 100. The values of n for the weakly bound H^+(L-Trp)(D-glucoside)$(N_2)_n$ clusters were $n \leq 4$ (maximum $n = 4$). It was reported that gas-phase N_2 adsorption-mass spectrometry could be used to detect the presence of free X–H (X = O and N) groups on the surface of gas-phase ions (Fujihara and Shimada 2019; Ochiai et al. 2020). N_2 molecules were adsorbed on the hydrogen atoms of the hydroxy groups

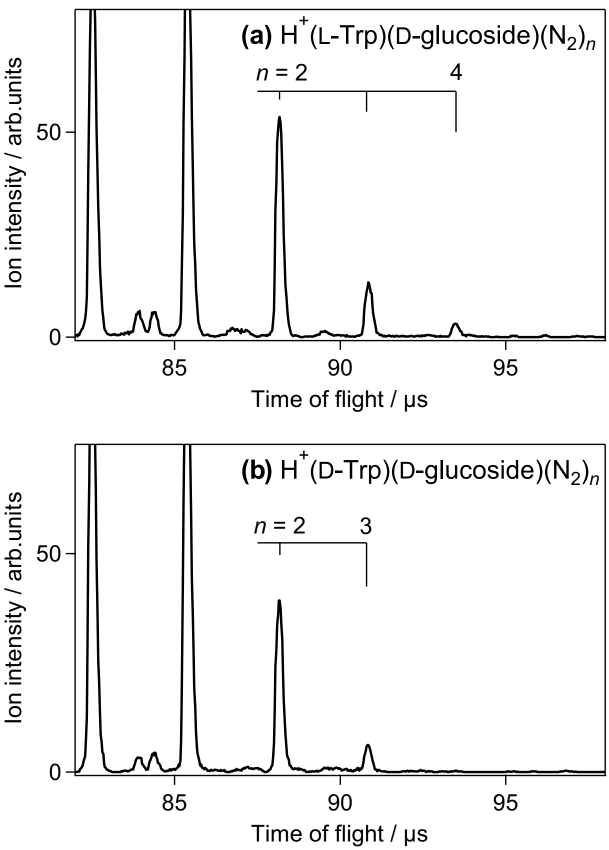

Fig. 2 Mass spectra of (**a**) H^+(L-Trp)(D-glucoside)$(N_2)_n$ and (**b**) H^+(D-Trp)(D-glucoside)$(N_2)_n$ formed via N_2 adsorption on mass-selected H^+(L-Trp)(D-glucoside) and H^+(D-Trp)(D-glucoside) at 50 K, respectively. The spectra were normalized by setting the ion intensities for $n = 1$ to 100

of carbohydrates, thereby facilitating quantification of the hydroxy groups of the carbohydrates via adsorption of gas-phase N_2 (Ochiai et al. 2020). $H^+Trp(N_2)_5$ is a stable magic number cluster wherein five N_2 molecules are adsorbed on the hydrogen atoms of the NH_3^+ group, indole ring, and carboxyl group (Fujihara and Shimada 2019). Therefore, the spectrum presented in Fig. 2a suggests that four free X–H groups is present on the surface of the heterochiral H^+(L-Trp)(D-glucoside) cluster.

Figure 2b illustrates the mass spectrum of H^+(D-Trp)(D-glucoside)$(N_2)_n$ formed via N_2 adsorption on homochiral H^+(D-Trp)(D-glucoside) at 50 K. The values of n for the weakly bound clusters formed in the ion trap were ≤ 3 (maximum $n = 3$). The relative ion intensity of the $n = 3$ cluster was lower than that of the heterochiral cluster, and the $n = 4$ cluster was not detected in the spectrum of homochiral H^+(D-Trp)(D-glucoside)$(N_2)_n$. The number of free X–H groups on the surface of homochiral H^+(D-Trp)(D-glucoside) is one less than that of heterochiral H^+(L-Trp)(D-glucoside). This indicates that the contribution of hydrogen bonds in the homochiral H^+(D-Trp)(D-glucoside) cluster outweighs that in the heterochiral H^+(L-Trp)(D-glucoside) cluster, given that the numbers and species of the functional groups containing the hydrogen-bonded clusters are the same.

Differences in the reactivity of various enantiomers in the cold gas-phase hydrogen-bonded clusters of chiral molecules were observed based on the product ion spectra acquired under 266 nm photoexcitation (Fujihara et al. 2014; 2017; Nguyen and Fujihara 2018;

Fujihara and Okawa 2018). The photoinduced cleavage of the C_α–C_β bond of cold gas-phase H^+(D-Trp) on a chiral crown ether was suppressed in the case of H^+(L-Trp) (Fujihara et al. 2014). In the case of H^+(D-Trp) on the chiral crown ether at 8 K, one of the hydrogen atoms of the NH_3^+ group was abstracted from the polyether ring, and the hydrogen atom induced C_α–C_β bond cleavage of D-Trp. In contrast, the three hydrogen atoms of the NH_3^+ group of H^+(L-Trp) at 8 K and H^+(D-Trp) at 300 K formed hydrogen bonds with the polyether ring, and C_α–C_β bond cleavage did not occur. Intermolecular hydrogen bonds between the chiral molecules suppressed the enantiomer-selective photodissociation.

Under the 266 nm photoexcitation of protonated Trp enantiomers that were hydrogen-bonded with D-sugars such as glucoside, glucose, galactose, talose, altrose, mannose, and maltose, cleavage of the C_α–C_β bond of D-Trp was suppressed in the homochiral clusters, whereas the cleavage of L-Trp occurred in the heterochiral clusters (Fujihara et al. 2017; Doan and Fujihara 2018; Nguyen and Fujihara 2018; Fujihara and Okawa 2018). This trend is consistent with the claim that the contribution of hydrogen bonds in homochiral H^+(D-Trp)(D-glucoside) exceeds that observed in heterochiral H^+(L-Trp)(D-glucoside), as deduced from the gas-phase N_2 adsorption data presented in Fig. 2.

Figures 3a and b illustrate the mass spectra of H^+(L-Trp)(D-ribose)$(N_2)_n$ and H^+(D-Trp)(D-ribose)$(N_2)_n$ formed via N_2 adsorption on H^+(L-Trp)(D-ribose) and H^+(D-Trp)(D-ribose) at 50 K, respectively. The values of n for the weakly bound clusters were $n \leq 3$, and the maximum number of adsorbed N_2 molecules was $n = 3$. The relative ion intensity of H^+(L-Trp)(D-ribose)$(N_2)_3$ was larger than that of H^+(D-Trp)(D-ribose)$(N_2)_3$, which was similar to the case of D-glucoside illustrated in Fig. 2. The relative ion intensities of the $n = 3$ clusters followed a similar

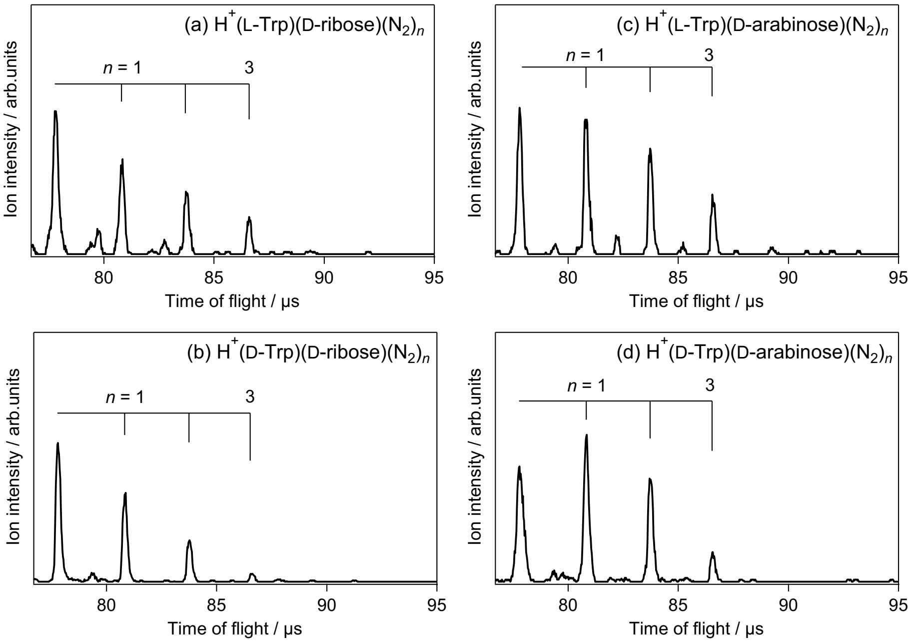

Fig. 3 Mass spectra of (**a**) H^+(L-Trp)(D-ribose)$(N_2)_n$, (**b**) H^+(D-Trp)(D-ribose)$(N_2)_n$, (**c**) H^+(L-Trp)(D-arabinose)$(N_2)_n$, and (**d**) H^+(D-Trp)(D-arabinose)$(N_2)_n$ formed via N_2 adsorption on mass-selected H^+(L-Trp)(D-ribose), H^+(D-Trp)(D-ribose), H^+(L-Trp)(D-arabinose), and H^+(D-Trp)(D-arabinose) at 50 K, respectively

trend in the case of D-arabinose, as illustrated in Figs. 3c and d. The ion intensity distributions of the weakly bound clusters formed via N_2 adsorption on the gas-phase ions indicated multiple structures of the gas-phase ions. Although the maximum number of N_2 molecules adsorbed on Na^+(glucose) was $n = 5$, the relative ion intensity of $Na^+(glucose)(N_2)_5$ was lower than that of the other hexoses containing five hydroxy groups (Ochiai et al. 2020). The lower relative ion intensity of the $n = 5$ cluster in the mass spectrum of glucose is ascribed to the formation of an intramolecular hydrogen bond in the structure via the hydroxy group. Therefore, the lower relative ion intensities of homochiral H^+(D-Trp)(D-ribose)$(N_2)_3$ and H^+(D-Trp)(D-arabinose)$(N_2)_3$ compared to those of the heterochiral clusters indicate that the contribution of hydrogen bonds in the homochiral clusters is larger than that in the heterochiral clusters, as in the case of glucoside.

Formation of Molecular Cloud on Hydrogen-Bonded Clusters

Water adsorption on the cold gas-phase hydrogen-bonded clusters was investigated to understand the relationship between heterogeneous nucleation and the chiral recognition of amino acids and monosaccharides in interstellar molecular clouds. Binding energy of water molecules to protonated Trp in the gas phase was larger than that of diatomic molecules

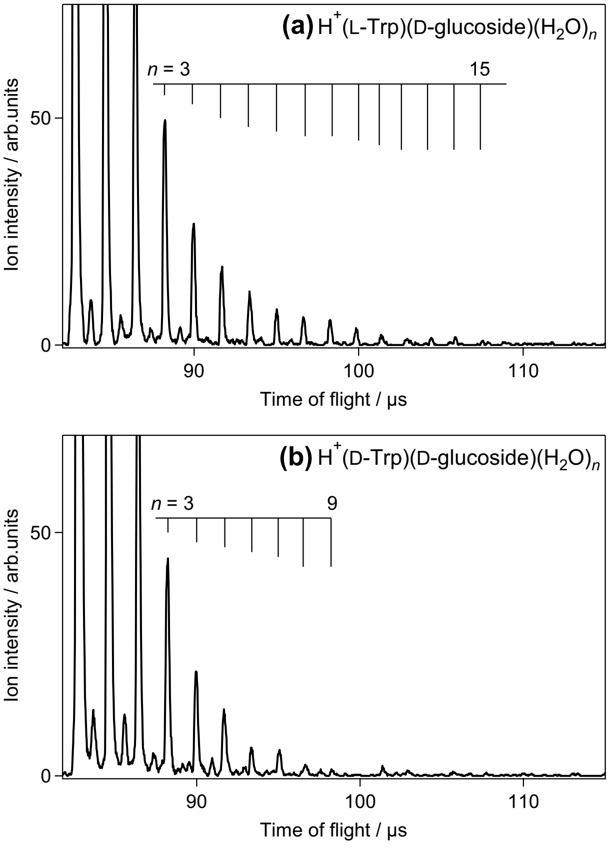

Fig. 4 Mass spectra of (**a**) H^+(L-Trp)(D-glucoside)$(H_2O)_n$ and (**b**) H^+(D-Trp)(D-glucoside)$(H_2O)_n$ formed via H_2O adsorption on mass-selected H^+(L-Trp)(D-glucoside) and H^+(D-Trp)(D-glucoside) at 8 K, respectively. The spectra were normalized by setting the ion intensities for n = 2 to 100

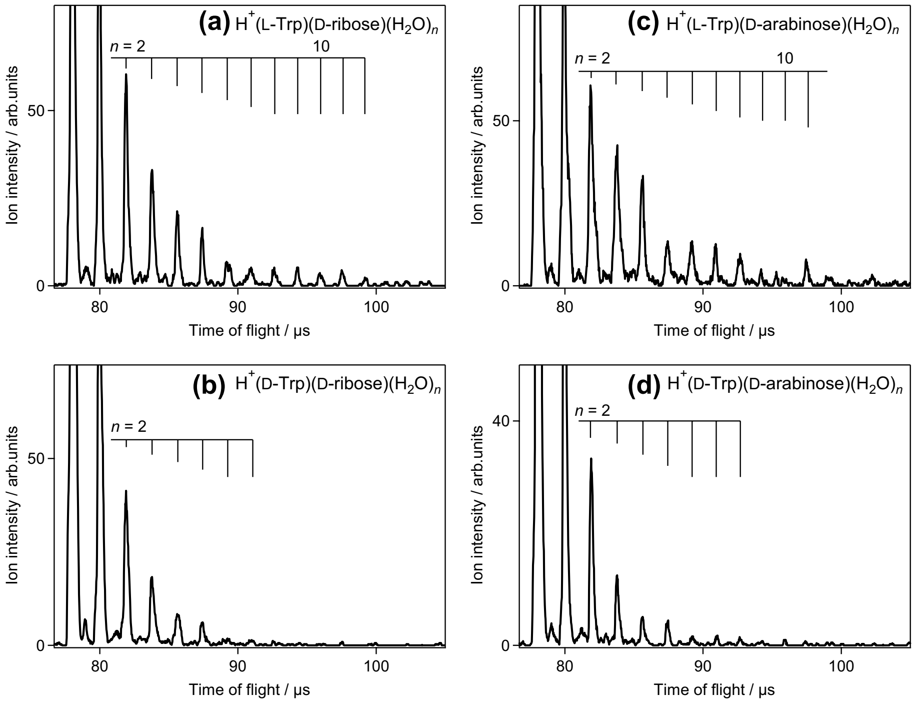

Fig. 5 Mass spectra of (**a**) H^+(L-Trp)(D-ribose)$(H_2O)_n$, (**b**) H^+(D-Trp)(D-ribose)$(H_2O)_n$, (**c**) H^+(L-Trp)(D-arabinose)$(H_2O)_n$, and (**d**) H^+(D-Trp)(D-arabinose)$(H_2O)_n$ formed via H_2O adsorption on mass-selected H^+(L-Trp)(D-ribose), H^+(D-Trp)(D-ribose), H^+(L-Trp)(D-arabinose), and H^+(D-Trp)(D-arabinose) at 8 K, respectively. The spectra were normalized by setting the ion intensities for $n = 1$ to 100

such as H_2 and N_2 (Spieler et al. 2018). Water molecules formed various hydrogen-bonding networks using their oxygen and hydrogen atoms in the gas phase (Miyazaki et al. 2004).

Figures 4a and b illustrate the mass spectra of H^+(L-Trp)(D-glucoside)$(H_2O)_n$ and H^+(D-Trp)(D-glucoside)$(H_2O)_n$ formed via H_2O adsorption on H^+(L-Trp)(D-glucoside) and H^+(D-Trp)(D-glucoside) at 8 K, respectively. The spectra were normalized by setting the ion intensity for $n = 2$ to 100. For the $n = 1$ heterochiral and homochiral clusters, the relative ion intensity was 180 and 240, respectively. Compared to the maximum value of 9 for homochiral H^+(D-Trp)(D-glucoside)$(H_2O)_n$, more than 15 H_2O molecules were adsorbed on heterochiral H^+(L-Trp)(D-glucoside). Larger water clusters were formed on the heterochiral clusters than on the homochiral clusters. Water adsorption on the cold gas-phase hydrogen-bonded clusters containing Trp enantiomers and D-monosaccharides such as D-ribose and D-arabinose was similar to that on D-glucoside, as illustrated in Fig. 5. Water molecules were preferentially adsorbed on the heterochiral clusters relative to the homochiral clusters.

The contributions of hydrogen bonds in the homochiral clusters of D-Trp and D-monosaccharides were larger than those in the heterochiral clusters, and the numbers of free X–H groups on the surface of the homochiral clusters were fewer than those on the surface of the heterochiral clusters. A hydrogen-bonding network of water molecules was preferentially formed on the surface of the cold gas-phase hydrogen-bonded heterochiral clusters containing L-Trp and D-monosaccharides.

For the chemical process in the cold gas-phase protonated clusters containing Trp enantiomers and D-monosaccharides, the enantiomer-selective photodissociation of L-Trp formed an excess of D-Trp (Fujihara et al. 2017; Nguyen and Fujihara 2018; Fujihara and Okawa 2018). For the physical process on the surface of the clusters, H_2O molecules were preferentially adsorbed on the heterochiral clusters containing L-Trp and D-monosaccharide in the gas phase. Enantiomer selectivities were different between in the cold gas-phase clusters and on the surface of the clusters.

Conclusions

The relationship between molecular adsorption and chiral recognition of amino acids and monosaccharides in the gas phase was investigated using a tandem mass spectrometer equipped with a cold ion trap. The adsorption sites on the surface of cold gas-phase hydrogen-bonded cluster ions were quantified using gas-phase N_2 adsorption-mass spectrometry. Water molecules were preferentially adsorbed on cold gas-phase heterochiral clusters containing L-amino acid and D-monosaccharide, and larger water clusters were formed on the surface of the clusters. Laboratory studies on physical and chemical properties of cold gas-phase hydrogen-bonded clusters containing biological molecules were useful for investigating enantiomer selectivity and chemical evolution in interstellar molecular clouds.

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