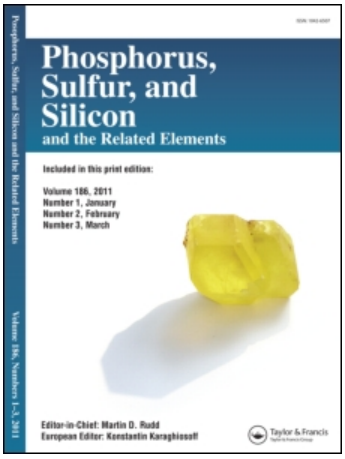

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ADSORPTION-DESORPTION CHARACTERISTICS OF WATER VAPOR ON FUNCTIONAL CATTLE BONE-ORIGINATED APATITES POWDERS WITH MESOPORES

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Microcrystals of cattle bone-originated apatites (r-HAp) were prepared by the dissolution-precipitation and freeze-drying processes. The r-HAp particles obtained gave 128 $m^2 \cdot g^{-1}$ specific surface area and 0.376 $cm^3 \cdot g^{-1}$ total pore volume and strong basic surface with mesopores. All the adsorption isotherms of water vapor at 288–308 K for the r-HAp powders obeyed IV-type in a classification of the BDDT. In the repeated adsorption-desorption operations, an adsorption hysteresis resulting from mesopores was recognized. The amounts of water vapor adsorbed obtained in the first adsorption operation were larger than those in the second adsorption operation, indicating that some portion of water vapor adsorbed to be irreversible adsorption. The water vapor-adsorption heats for the r-HAp were 45–60 $kJ \cdot mol^{-1}$, whose values were higher than natural diatomite. The amounts of water vapor adsorbed for the r-HAp were larger than those for the adsorbents, such as natural diatomite, aerosol silica, and activated carbon, at the relative partial pressures of 0–0.7.

Keywords: Cattle bone; hydroxyapatite; mesopore; particle design; self-humidity control; water vapor-adsorption

INTRODUCTION

For solving the serious problems on human living environments, such as dew condensation, putrefaction bacteria, and static electricity, some

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self-humidity conditioning materials, which have high specific surface areas and a lot of mesopores, have been developed.[1,2] Based on correlations between the solid surfaces developed and water vapor-adsorption characteristics, it was demonstrated that the mesopores with 4–20 nm on the surfaces would be effective for excellent self-humidity conditioning ability. Hydroxyapatite (HAp: $Ca_{10}(PO_4)_6(OH)_2$), which has good biocompatibility and adsorption properties for biopolymers, is one of the nano-crystal materials with mesopores. The HAp powders have been applied as column packing materials for liquid chromatography to the separation of proteins and nucleic acids.[3]

Cattle bone-originated apatites (r-HAp) prepared by a dissolution-precipitation method have a structure-sensitive surface with mesopores.[4,5] The r-HAp particles can easily be modified in the number and nature of active sites on the surfaces, as well as pore structures by using the specified drying procedures.[6] The powders prepared thus can be applied to drying or humidity control agents for biomaterials.

The aims of this study are to characterize the surface structures of the r-HAp powders fabricated by a freeze-drying technique and to investigate the adsorption-desorption behavior for water vapor on the surfaces by comparing them with some commercial adsorbents.

EXPERIMENTAL PROCEDURE

Cortical bones in cattle femur (Holstein bull, Hokkaido prefecture, Japan) were calcined and dissolved to an HNO_3 solution and reprecipitated at 298 K and pH 10.5 for 24 h.[4,5] The r-HAp particles with a single phase were fabricated by the freeze-drying technique. For the characterization of the r-HAp particles obtained, they were observed by a scanning electron microscope (SEM) to reveal the morphology and size. The BET specific surface area and pore size distribution of the particles were measured by N_2-adsorption at 77 K. The acid and basic properties of the surfaces were evaluated by a temperature programmed desorption of CO_2.

Characterizing the adsorption-desorption behavior of water vapor, the sample powders were dried at 393 K for 24 h in air and evacuated at 473 K for 5 h in vacuum. The amounts of water vapor adsorbed were measured at 288–308 K under the relative partial pressures (P/Ps) of 0–0.95 by an automatic water vapor-adsorption isotherms apparatus (Japan Bell Co.). The adsorption heats of water vapor were calculated

from the adsorption isotherms obtained at 288–308 K by using the Clausis-Clapeyron equation.[4,5]

RESULTS AND DISCUSSION

The freeze-drying process produced the frock like-particles having 5–10 μm in size, which consisted of about 100 nm microcrystals. The r-HAp particles exhibited 128 $m^2 \cdot g^{-1}$ specific surface area and 0.376 $cm^3 \cdot g^{-1}$ total pore volume. In the analysis of the pore size distribution curves, the pore volumes fell between the pore diameters of 2–20 nm. The basicity of the r-HAp surfaces with mesopores was stronger than that of the synthetic HAp surfaces prepared from reagents.

Figure 1 shows adsorption isotherms of water vapor at the adsorption temperatures of 288–308 K for the r-HAp powders. For each of the adsorption isotherms, the amounts of water vapor adsorbed increased through raising the absolute partial pressure (P) to the saturated water vapor-pressures (Ps) and decreased with diminishing the P value. In the repeated adsorption-desorption operations, one can recognize that an adsorption hysteresis resulted from mesopores. From these results, it was suggested that the adsorption isotherms of water vapor obeyed IV type in a classification of the BDDT.

Figure 2 shows first and second adsorption isotherms of water vapor at 298 K for the r-HAp powders. The second isotherm was measured in the same condition of the first isotherm after the evacuation at 295 K for 24 h in vacuum. The amounts of adsorbed water vapor obtained in the first adsorption operation were larger than those in the second adsorption operation. This difference in the amounts of adsorption-desorption means that some portion of adsorbed water vapor includes irreversible

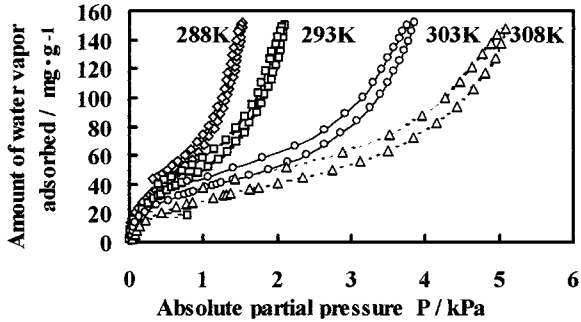

FIGURE 1 Water vapor-adsorption isotherms at different temperatures for the r-HAp powders freeze-dried.

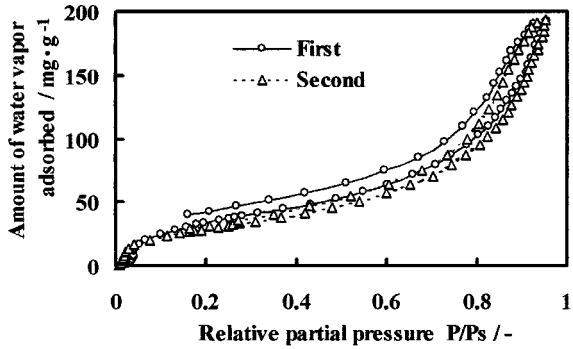

FIGURE 2 Water vapor-adsorption isotherms at 298 K for the r-HAp powders freeze-dried.

adsorption. But for natural diatomite (NDM) powders, which have been known as physical adsorption, the second adsorption isotherm of water vapor was the same as the first one. The adsorption heats of water vapor for the r-HAp were 45–60 $kJ \cdot mol^{-1}$, which were 2–3 times higher than those for the NDM.

Figure 3 shows water vapor-adsorption isotherms at 298 K for various commercial adsorbents. The amounts of water vapor adsorbed for the r-HAp were larger than those for the NDM aerosol silica (AE200), and activated carbon (AC) at the P/Ps of 0–0.7.

Based on these results presented, it was found that the freeze-dried r-HAp powders had effective surfaces in mesopores for self-humidity conditioning ability.

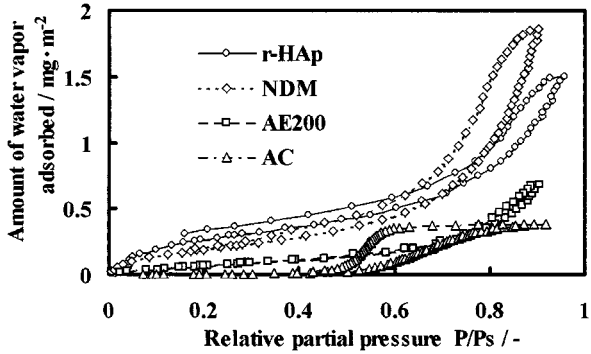

FIGURE 3 Water vapor-adsorption isotherms at 298 K for different adsorbents.

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