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한국 서해 초기현세 퇴적물중 자생 능철석의 원소 성분과 퇴적학적 의미

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Elemental Composition of Authigenic Siderites in the Early Holocene Coastal Sediments, Western Coast of Korea and Their Depositional Implication

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Abstract: Authigenic siderite grains, ranging 100 to 250-μm in diameter, are abundant in an about 8,600-year-old sediment layer in Namyang Bay, west coast of Korea. The siderites exhibit the aggregated spherulitic morphology with well-developed rhombs on the grain surfaces. They consist mostly of $FeCO_3$ (average, 65%) and $MnCO_3$ (average, 22%) with low Mg/Ca ratio (less than 0.4) in their bulk composition. A series of compositional ternary discrimination diagrams, together with high Mn and low Mg contents, show that only meteoric porewater was involved in siderite precipitation, assuming that depositional environment of host sediment is an organic-rich freshwater system. Considering a series of results such as radiocarbon age, authigenic Mn-rich siderite and lithological features, siderite-hosting sediment (unit T1) is interpreted as freshwater swamp or bog deposition, infilling the topographic depressions that locally existed before the formation of mid-to-late Holocene tidal deposits. Center-to-margin compositional variation within individual grain is very systematic; Mn and Ca decrease towards the margin of a siderite grain, while Fe and Mg increase. It suggests that the spherulitic siderites were precipitated in this sedimentary layer in a series during the early diagenesis of MnOx-FeOx reduction under steady-state.

Keywords : spherulitic siderite, freshwater environment, early diagenesis, west coast of Korea

요 약 : 한국 서해 남양만 조간대의 초기현세 (ca. 8,600 yr BP) 니질 퇴적물에서 특징적으로 조립사 (100~250 μm) 크기의 자생 능철석 입자들이 풍부하게 발견된다. 이들 갈색의 능철석 입자들은 표면에 잘 발달된 육면체 결정을 가진 구형의 집합체 형태를 보이며, 주성분 원소는 철과 망간으로 각각의 평균 함량이 각각 65%와 22%에 이른다. 높은 망간함량과 낮은 마그네슘 함량 그리고 성분삼각도표 (compositional ternary discrimination diagram) 결과들은 이들 능철석 입자들이 담수환경에서 형성되었음을 보여준다. 결과적으로 능철석 입자들을 함유한 초기현세 퇴적물 (unit T1)은 해침의 초기 단계에서 해수면과 일정 거리를 가진 해안선 근처의 육지에 발달한 소규모 담수 습지 (freshwater swamp or bog) 퇴적층으로 해석된다. 한편, 능철석 입자 내에서 뚜렷한 화학적 성분 변화가 나타나는 바, 입자의 중심에서 바깥쪽으로 갈수록 철과 마그네슘의 함량은 증가하는 반면 망간과 칼슘의 함량은 감소하는 경향을 보인다. 이러한 결과는 자생 능철석 입자들이 퇴적층의 공극내에서 일련의 초기속성작용 (early diagenetic process) 동안 순차적으로 형성되었음을 반영한다.

주요어 : 구상의 능철석, 담수환경, 초기속성작용, 한국서해연안

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Introduction

Authigenic siderites have been described from a variety of sediments, sedimentary rocks, and palaeosols (Postma, 1977; Gautier, 1982; Bahrig, 1989; Browne and Kingston, 1993; Baker *et al.*, 1996; Fisher *et al.*, 1998). They generally form under highly reducing conditions characterized by high carbonate and low sulfide concentrations (Pye, 1984; Curtis *et al.*, 1975; Pearson, 1979; Postma, 1982; Curtis *et al.*, 1986). Thus, the most favorable environment for siderite formation seems to be the organic-rich, reduced freshwater environments such as lake, swamp, and marsh (Postma, 1981 and 1982; Bahrig, 1989; Zodrow *et al.*, 1996; Khim *et al.*, 1999). Siderite formation from sea-water environments is restricted because of the stronger affinity of Fe^{2+} with HS^- ions than with HCO_3^- ions. However, where iron(III) reduction exceeds sulphate reduction, iron-rich carbonate minerals may co-precipitate with iron sulphide (Coleman, 1985; Curtis *et al.*, 1986). Consequently, siderite formation is largely controlled by the chemistry of porewater, resulting in difference in the elemental composition of siderite. Mozley (1989) established the similar relationship between siderite composition and the composition of the water from which it precipitated. Because of this, geochemistry of siderite is potentially a useful characteristic for palaeoenvironmental study of sedimentary deposits for abundant organic-matter-bearing sediments (Mozley, 1989; Mozley and Wersin, 1992; Khim *et al.*, 1999).

Park *et al.* (1995) noted the occurrence of siderite concretions in early Holocene sediments from Namyang Bay, but detailed information on their morphology, elemental compositions, and mode of formation are not available. An important aspect of this occurrence is that the paleoenvironment of the siderite-bearing sediment cannot be determined adequately owing to an absence of diagnostic sedimentological information (Park *et al.*, 1995). Siderite may provide insight into the depositional environment in Namyang Bay during the Holocene sea-level rise. The aim of this paper is to interpret the origin of siderite and depositional environments of siderite-hosting sediment based on the textural and morphological features and elemental com-position of siderite grains.

Study Area and Stratigraphic Framework

Namyang Bay, located on the west coast of Korea, is one of the major ria-type coastal embayments found in Korea (Fig. 1). The bay is a large, east-west-oriented, funnel-shaped embayment that is 12km long, 3~9km wide, and penetrates deeply into the land. Well-defined seaward barriers, rivers, and otherwise typical salt marsh systems are lacking in this bay. The tides are semi-diurnal, with a mean tidal range of 5.7m (spring, 8.3m; neap, 4.9m, Kim, 1989). There is a wide muddy tidal flat along the southern shoreline of the bay with average widths of 2~5km (Fig. 1). A large, intertidal bank is located in the northern subtidal channel. The tidal flat drainage system consists of 10 intertidal channels, which are essentially stable, showing little tendency for lateral migration (Alexander *et al.*, 1991). This Namyang Bay tidal flat is typical of the muddy tidal flats that are common along much of the west coast of Korea (Wells and Huh 1979; Wells *et al.*, 1990; Alexander *et al.*, 1991).

Three late Quaternary units are present in the Namyang tidal-flat deposit (Park *et al.*, 1995): an upper marine mud (unit M1), a middle siderite-hosting terrestrial clay (unit T1) and a lower marine mud (unit M2) (Fig. 2). The uppermost deposit (unit M1) is up to 7 m, consists of soft gray or greenish gray mud, sandy mud, and sand. This unit represents a typical Holocene intertidal mudflat deposit, showing a coarsening-upward textural trend that has resulted from the continual retrogradation of tidal flat during the mid-to-late Holocene sea-level rise (Park *et al.*, 1995; Kim *et al.*, 1999). Unit T1 is separated from unit M1 by an unconformity, and consists of reddish brown and homogeneous clay

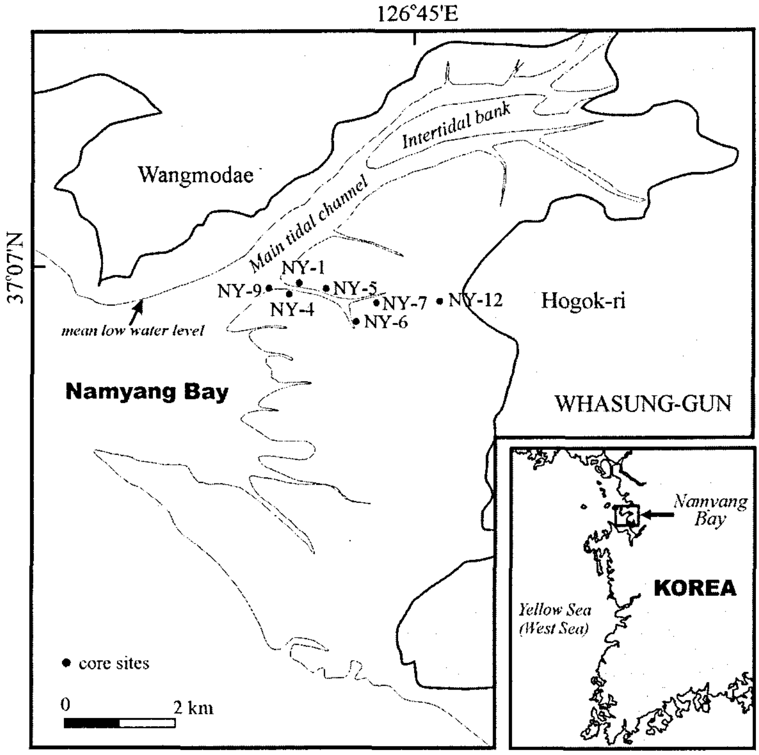

Fig. 1. Index map showing Namyang Bay, western coast of Korea (eastern Yellow Sea) and enlarged map of study area and core locations on the intertidal flat of the bay.

sediments. A radiocarbon age for the organic debris in this unit is 8,597±77yr BP (Park *et al.*, 1995), interpreted to be a time of Holocene transgression, but before marine flooding had reached the study area (Bloom and Park, 1985; Cang *et al.*, 1997). In particular, this unit contains considerable amounts of siderite grains, pellets and organic debris (Park *et al.*, 1995). Compared to extensive, well-constrained studies of unit M1 deposits, studies of early Holocene deposits are relatively rare. Although this radiocarbon age of this unit and comparison with a sea-level curve indicate that unit T1 is terrestrial deposition, a more comprehensive interpretation for depositional environment of this unit is required. Unit M2 is separated from unit T1 by an unconformity, and consists of yellowish orange muddy sediments characterized by semi-consolidation and cryogenic structures, suggesting a significant degree of weathering during the sea-level lowstand (Park *et al.*, 1995; Lim, 2001). This unit can be correlated with the upper part of the late Pleistocene tidal deposits reported ubiquitously along the west coast of Korea (Park *et al.*, 1995; Park *et al.*, 1998; Lim, 2001; Lim *et al.*, 2002).

Analytical Methods

The Early Holocene stratigraphic sediment sections in several Namyang-Bay cores were logged in detail on the basis of visual examination, and X-radiography and grain size analyses for these sediments were performed by standard sieving and pipetting method. Shear strength was gauged by using a Torvane Rheometer at about 10cm intervals, and water contents of sediments were also measured at same intervals. The particulate organic carbon (POC) content of sediments was measured using the back-titration method (Alison, 1965).

After routine grain-size analysis without any chemical treatment, dark-brown-colored mineral grains

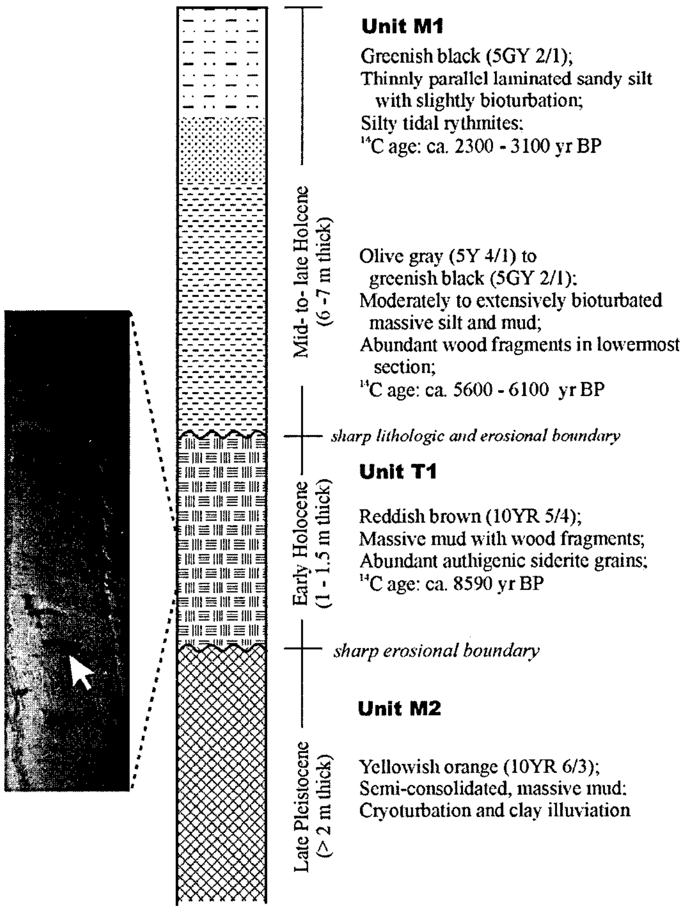

Fig. 2. Schematic stratigraphic overview in the late Quaternary Namyang tidal-flat deposits (after Park *et al.*, 1995).

were hand-picked from sand fractions under a stereomicroscope. The grains were powdered for X-ray diffraction analysis, and impregnated with low-viscosity epoxy resin for polished-thin sections. A sideritic composition was confirmed by X-ray diffraction analysis, and elemental compositions were analyzed quantitatively using a JEOL JXA-8900R electron microprobe equipped with a wave-dispersive spectrometer (WDS). All analyses were performed with a 5-μm beam width, 10-nA sample current, and 15-kV acceleration potential, using carbonate standards. A ZAF correction was applied to the microprobe analysis. In particular, center-to-margin compositional variation of some grains was analyzed at 5 to 10-μm intervals. To analyze the compositional variations in each grain, CaKαX-ray mapping was done with an automated JEOL JXA-8900R electron microprobe, using a 1-μm beam diameter. Also, the detailed morphology and internal texture of the grains were delineated using a scanning electron microscope (SEM, JEOL JSM840A).

Results and Discussion

Occurrence, Morphology and Elemental Composition of Siderite

Siderite grains are abundant in early Holocene sediment (unit T1), which is characterized by reddish- or yellowish-brown (10YR 5/4) massive mud, consisting of 53% silt, 45% clay, and less than 5% sand. Primary sedimentary structures are not readily

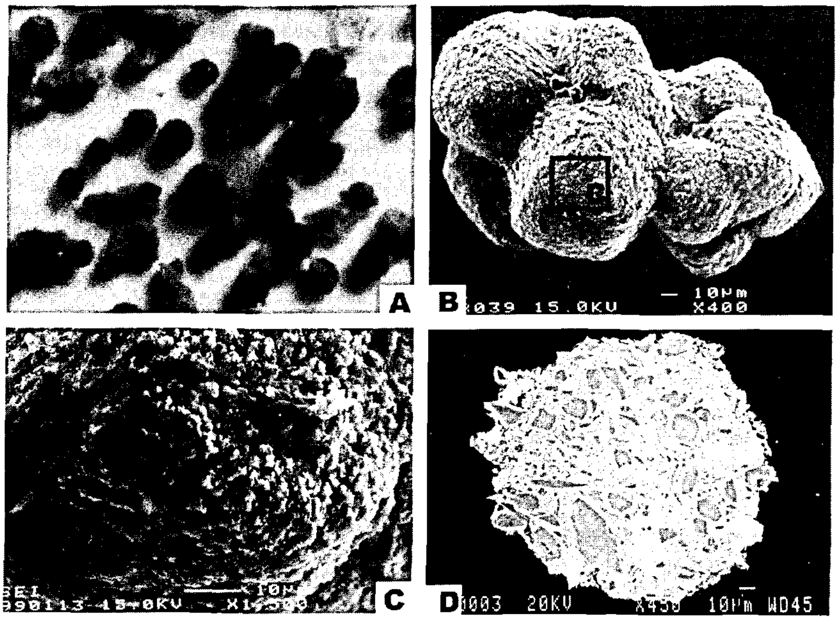

Fig. 3. (A) Siderite grains obtained from 2phi fraction of the reddish brown sediment (unit T1) of Namyang Bay. (B) SEM photograph showing a typical spherulitic siderite grain. (C) SEM photograph of texturally well-developed rhombs on the grain surface. (D) Backscattered electron image of siderite showing many inclusions composed mostly of quartz, feldspar and other clay minerals.

discerned, perhaps because burrows created by dwelling and feeding activities of benthic organisms are common. Wood fragments are ubiquitous throughout this unit and thus the sediments are relatively enriched in organic matter, having higher organic carbon (0.5~0.7%; 0.6% in average) than that of the overlying Holocene marine tidal sediments (0.2~0.4%). Such fairly high contents of organic carbon are a prerequisite for precipitation of authigenic minerals. Average water content is 30% (ranges from 27% to 36%) and shear strength is 3.2kg/cm^2 (ranges from 2.2kg/cm^2 to 5.6kg/cm^2).

Siderite grains are typically spherulitic nodules, ranging from 100 to 250-μm in diameter, that can be readily identified by their dark yellowish-brown color and spherical shape (Fig. 3a). Siderite grains with comparable size and shape have been reported from other coastal sediments (Zhang *et al.*, 1996; Khim *et al.*, 1999). Most of the siderite grains make groups forming band- or chain-shaped aggregates, which are up to several centimeters long (Fig. 3b). The aggregates can be easily separated into individual grains during mechanical grain-size analysis. SEM images show that each siderite grain is spherical and has distinct rhombs on its surface (Fig. 3c). Back-scattering images (BSI photographs) show that the internal structure is homogeneous, lacks any concentric structure, and consists of an

Table 1. Percentage ranges and mean values of elemental compositions for five siderite grains.

Grain No.	Mean & Range	Fe (%)	Ca (%)	Mg (%)	Mn (%)
NY-1	Mean	63.2	10.6	3.2	23.0
	Min.	53.7	6.7	0.9	18.0
	Max.	69.4	14.8	10.7	30.8
NY-2	Mean	66.1	7.9	4.1	21.9
	Min.	62.6	6.7	1.9	19.4
	Max.	69.4	9.7	6.8	24.9
NY-4	Mean	60.7	11.7	3.3	24.3
	Min.	53.7	7.4	0.9	19.5
	Max.	65.7	14.8	10.7	30.8
Ny-12	Mean	68.0	9.5	2.3	20.2
	Min.	66.3	8.0	1.2	18.0
	Max.	68.9	10.7	6.9	22.7
Average		64.5	9.9	3.2	22.4

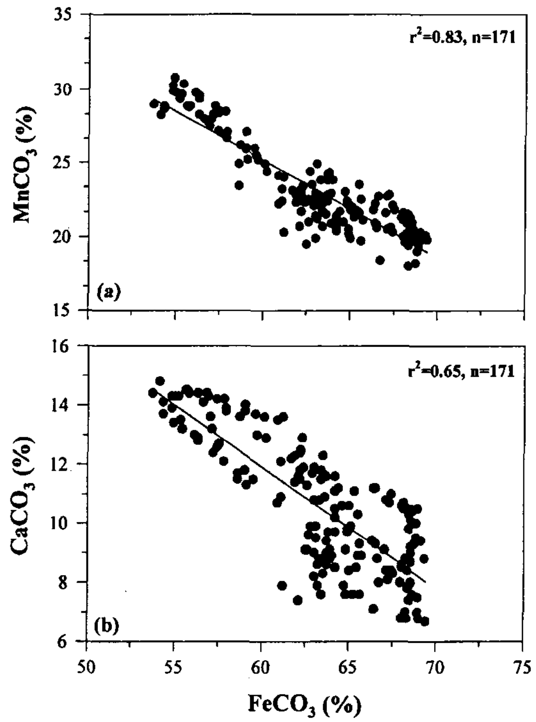

Fig. 4. Correlation between $FeCO_3$ and $MnCO_3$ (A) and $FeCO_3$ and $CaCO_3$ (B).

aggregation of minute crystals (Fig. 3d). Quartz grains and other impurities are commonly scattered randomly throughout the siderite grain (Fig. 3d). The impurities are identified as diverse clay-sized minerals (e.g., illite, albite etc.) by WDS and XRD analyses.

Siderite composition (Table 1) is 54~69mol% $FeCO_3$ (average 65%), 18~31mol% $MnCO_3$ (average 22%), and 7~15mol% $CaCO_3$ (average 10%). $MgCO_3$ contents range from 1 to 11mol% (average 3%), but are typically less than 4mol% $MgCO_3$. There are negative good linear relationships between Fe and Mn content ($r^2 = 0.83$) and Fe and Ca content ($r^2 = 0.65$) (Fig. 4). Compositional differences of each grain are minor, but considerable within an individual concretion. Figure 5 shows the chemical variability within a siderite concretion. $FeCO_3$ contents, relatively low in the center of the grain (54mol%), largely increase towards the margin (66mol%); contrastingly, $MnCO_3$ and $CaCO_3$ contents decrease. $MgCO_3$ contents, as low as 1mol% in the center, also increase towards the margin of the siderite (6mol%). $MnCO_3$ contents systematically decrease from 30mol% in the center of the grain to 20mol% in the margin (Fig. 5). Similarly, $CaCO_3$ contents are only 9mol% in the margin, while as high as 15mol% at the center. In the outmost layer, $FeCO_3$ and $MnCO_3$ contents appear to be decreased, while $MgCO_3$ and $CaCO_3$ contents increase. These compositional variations can be seen clearly in the image mapping data, analyzed at 1-μm intervals (Fig. 6).

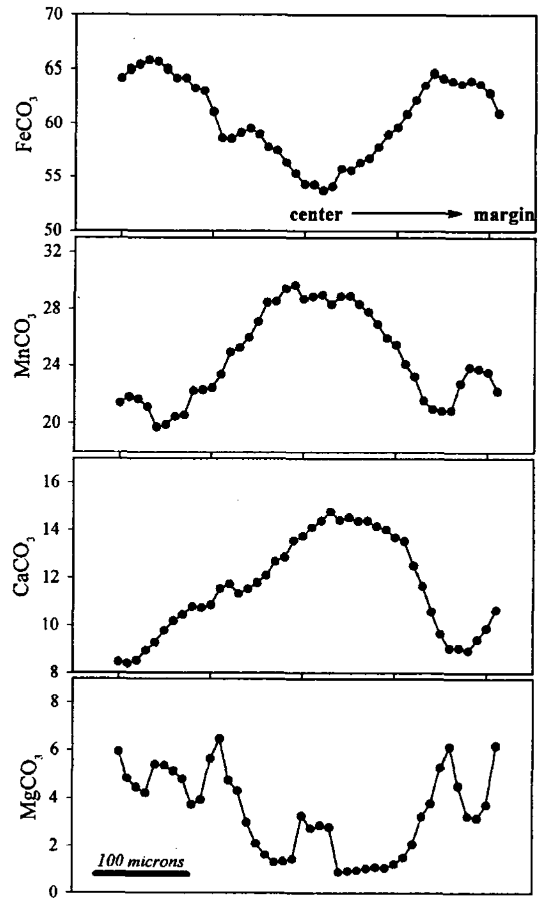

Fig. 5. Compositional variation within selected siderite grains. Note the systematic center-to-margin variations.

Depositional Implication and Origin of Siderite

The siderites in unit T1, a typical single spheru-

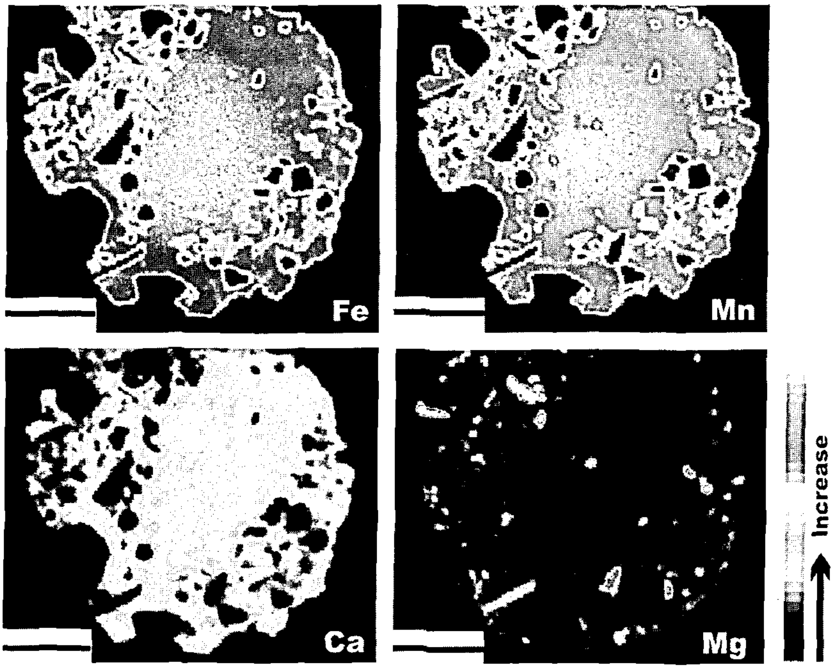

Fig. 6. X-ray maps of siderite grain. Overall, intensities of Mn and Ca decrease from center to margin, but intensity of Fe increases. Scale bar is 20 micorns.

litic grain (equivalent to spherosiderite) in morphology without cracks (Fig. 3d), are similar in their physical characters to those in the swamp deposit of Kyunggi Bay (Khim, *et al.*, 1999) and the palaeosol of Boss Point Formation, Maritime Canada (Browne and Kingston, 1993). Pearson (1979) and Postma (1982) suggested that the spherulitic siderite would have formed authigenically in the soft muddy sediments of small-localized terrestrial environments (e.g., stagnant pond, bog, and swamp) characterized by abundant organic materials and low dissolved sulfate concentration. In addition, lack of any cracks within the siderite grains implies that they were mostly formed by direct precipitation during the early diagenesis of sediments (Moore *et al.*, 1992; Khim *et al.*, 1999).

Mozley (1989) reported that early diagenetic siderites from marine sediments are always extremely impure in their chemical composition characterized by the extensive substitution of Mg and Ca for Fe as well as by a relatively high Mg/Ca ratio. Compared to marine siderite, however, freshwater siderites are pure, high in Mn content (greater than 2mol% $MnCO_3$), and low in Mg/Ca ratio (Mozley, 1989). Although the siderites from the study area are slightly depleted in Fe content compared to those from the other areas, all data fall within the zone characterizing the formation of freshewater siderite (Fig. 7). The mean ionic Fe : Ca : Mg ratios are 82 : 14 : 4, indicating that the siderites are pure in their composition compared to other marine siderites showing more variations in Ca and Mg contents (Fig. 7a). In the Ca-Mg-Mn ternary diagram (Fig. 7b), most of the data points are also grouped well (29 : 9 : 62) and plotted between the extremes of Mn and Ca. These diagrams show that the siderite samples of the study area are enriched in Mn and Ca contents, while largely depleted in Mg content, resulting in low Mg/Ca ratios. In a series of com-positional ternary discrimination diagrams (Fig. 7), the Namyang-Bay siderites have comparable compositions to the non-marine field of Mozley (1989), having enriched Fe and Mn components than the marine field. It indicates that siderite-host-

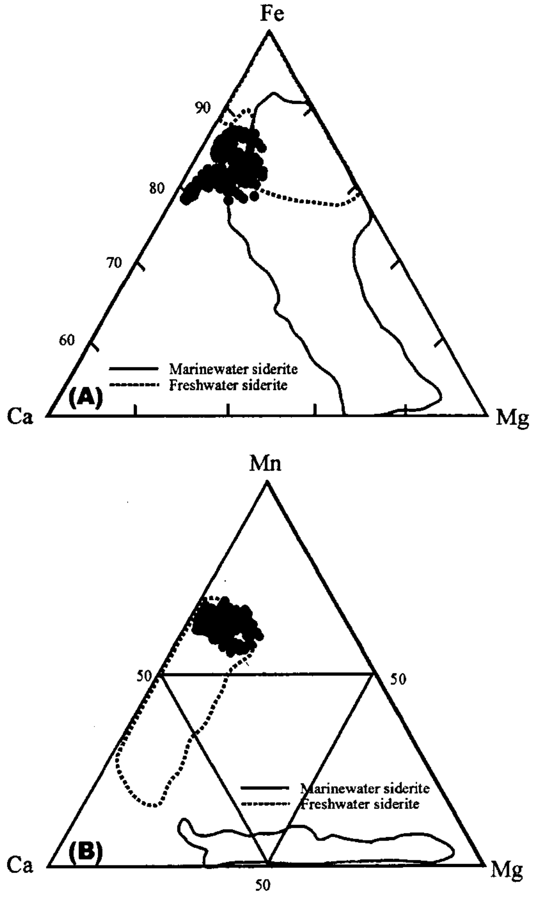

Fig. 7. Compositional ternary discrimination diagrams for freshewater and marine siderites (after Mozley, 1989 and Khim *et al.*, 1999). (A) Fe-Ca-Mg and (B) Mn-Ca-Mg diagrams. Most of data fall within the freshewater siderite field.

ing sediment (unit T1) is freshwater deposition, such as swamp or bog environments generally characterized by abundant organic matter, very fine-grained clay matrix and low level of sulfate. Furthermore, wood fragments (approximately 2m below mean sea level) obtained from this unit were dated at 8,597± 77yr BP. At this time, the sea level on the west coast of Korea was at least 10~20m below the present mean sea level (e.g., Bloom and Park, 1985; Chang *et al.*, 1996). This fact indicates that unit T1 sedi-ments might have been deposited above the sea level at that time. Considering a series of results such as the presence of authigenic siderite grains, radiocarbon age, and lithologic features, consequently, unit T1 is interpreted as early Holocene non-marine sediments (e.g., swamp or bog deposition). By roughly 8~9ka BP, the Holocene transgression had not yet reached the present-day tidal flat, while the warm, humid climate at that time favored the formation of a freshwater swamp in the topographic lows where fine-grained sediments and organic debris accumulated.

As shown in Figures 5 and 6, an individual siderite grain shows a compositional variation from center to margin. At the centers of all individual grains, Mn contents are remarkably high, providing unequivocal evidence that the siderite precipitated close to the sediment-water interface and Mn and Fe were supplied by reduction of manganese and ferric oxides, as suggested by Curtis *et al.*, (1986). In particular, the decrease of Mn/Fe ratios toward the margin of a siderite grain is possibly due to the difference of reduction potentials between MnOx and FeOx with burial depth. This explanation is consistent with results of modern porewater studies of both marine and non-marine environments, which show that porewaters may become greatly enriched in Mn at very shallow depths. In the porewaters of modern marine and nonmarine sediments, generally, Fe_2^+ concentration frequently reaches a maximum in somewhat deeper depth than Mn_2^+. That is, early porewater was high in Mn_2^+ ion concentration and, with time or slightly deeper burial, its availability decreased relative to that of Fe_2^+ ions (Emerson, 1976; Froelich *et al.*, 1979; Shaw *et al.*, 1990). So, Mn contents in the siderite are decreasing while Fe contents are increasing. This zone (inner part of siderite grain) is comparable with the "early siderite formation stage" suggested by Curtis *et al.* (1986).

Mg content increases towards the margins of the siderite within all of the grains studied, although this general pattern is complicated by the growth of late Mn-rich overgrowths (Fig. 5). It seems likely that increases in the ratio of Mg to Fe within porewaters would cause an increase in the Mg substitution within siderite. Mg_2^+ ions could be supplied to the porewater from unstable Mg-bearing alumino-silicate minerals by the ion-exchange (clay mineral transformation) (Pearson, 1985; Curtis and Coleman, 1986). In this study area, smectite and chlorite contents are decreasing with depth as the lowering of pH in sediments (Lim, 2001), suggesting the possible supply of Mg ions for siderite precipitation by clay transformation. Although the increase of Mg/Fe ratio from center to margin could be due to the infiltration of overlying sea water by subsequent marine transgression (eg., Boles, *et al.*, 1985), siderites in this study do not seem to be subjected to sea water considering their very low Mg contents. This zone (outer part of siderite grain) is comparable to "late siderite" of Curtis *et al.* (1986). This result for the growth of siderite concretions provides unequivocal value for understanding the diagenetic environment in which Mn-rich siderite precipitates and also permits an assessment of the passive-infill mechanism suggested by mechanism in which siderite passively replaces porewater (Curtis *et al.*, 1986).

Conclusions

Authigenic siderites, occurring in the early Holocene sediments (unit T1) of Namyang tidal flats, are concluded to be formed in the freshwater environment, based on their pure bulk compositions with relatively high Mn and low Mg/Ca rations. This idea can be strongly supported by the age (ca. 8,597yr BP) of wood fragments collected in the siderite-hosting unit T1 where the sea-level was not reached to the unit T1. Systematic center-to-margin compositional variations in a siderite grain may be explained by in-a-series early diagenetic processes of MnOx-reduction, FeOx-reduction, and fermentation. Furthermore, the sequential growth model suggests that the siderite began to form during shallow burial and did not displace detrital grains.

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