Research Article

Material Exchange and Migration between Pore Fluids and Sandstones during Diagenetic Processes in Rift Basins: A Case Study Based on Analysis of Diagenetic Products in Dongying Sag, Bohai Bay Basin, East China

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The exchange and migration of basin materials that are carried by pore fluids are the essence of diagenesis, which can alter physical properties of clastic rocks as well as control formation and distribution of favorable reservoirs of petroliferous basins. Diagenetic products and pore fluids, resulting from migration and exchange of basin materials, can be used to deduce those processes. In this study, 300 core samples from 46 wells were collected for preparation of casting thin sections, SEM, BSE, EDS, inclusion analysis, and isotope analysis in Dongying Sag, Bohai Bay Basin, East China. Combined with geochemical characteristics of pore fluids and geological background of the study area, the source and exchange mechanisms of materials in the pore fluids of rift basins were discussed. It was revealed that the material exchange of pore fluids could be divided into five stages. The first stage was the evaporation concentration stage during which mainly Ca^{2+} , Mg^{2+} , and CO_3^{2-} precipitated as high-Mg calcites. Then came the shale compaction stage, when mainly Ca^{2+} and CO_3^{2-} from shale compaction water precipitated as calcites. The third stage was the carboxylic acid dissolution stage featured by predominant dissolution of plagioclases, during which Ca^{2+} and Na^+ entered pore fluids, and Si and Al also entered pore fluids and then migrated as clathrates, ultimately precipitating as kaolinites. The fourth stage was the organic CO_2 stage, mainly characterized by the kaolinization of K-feldspar as well as dissolution of metamorphic lithic fragments and carbon cements. During this stage, K^+ , Fe^{2+} , Mg^{2+} , Ca^{2+} , HCO_3^- , and CO_3^{2-} entered pore fluids. The fifth stage was the alkaline fluid stage, during which the cementation of ferro-carbonates and ankerites as well as illitization or chloritization of kaolinites prevailed, leading to the precipitation of K^+ , Fe^{2+} , Mg^{2+} , Ca^{2+} , and CO_3^{2-} from pore fluids.

1. Introduction

Pore fluids mainly refer to all fluids that occupy and pass through the pore space of sedimentary basins [1, 2]. During the burial process, basin materials could be loaded and redistributed by pore fluids. Those processes could lead to regularly distributed diagenetic products in basins [3]. Formation and evolution of diagenetic products could result in preservation or destruction of primary pores as well as formation and transformation of secondary pores, which could significantly influence the formation and occurrence of effective reservoirs in the deep part of petroliferous basins

[4–16]. Previous studies mainly highlighted evolution of physical properties of clastic rocks and primarily investigated diagenetic facies and sequences based on sedimentological and petrological analyses [11, 17–26]. However, insufficient attention was paid to the nature of diagenesis, namely, the exchange and migration of basin materials loaded by pore fluids. Meanwhile, many controversies arose concerning the formation mechanism of secondary pores, which further obscured prediction of distribution of secondary pores and effective reservoirs [5, 14, 23, 27–34]. Therefore, it should be critical to figure out the mechanism of basin material exchange and migration during the diagenetic process so as to

analyze the formation and distribution of diagenetic products and favorable sandstone reservoirs.

During diagenetic process, matrix of clastic rocks could be transformed or dissolved, and materials might partly or totally enter into pore fluids as ions or complexes. Solutes (ions or complexes) carried by pore fluids could be deposited as cements under appropriate geological conditions. In these processes, compositions of clastic rocks and chemical properties of pore fluids could be changed. These changes could be used to infer material exchange and migration mechanisms during diagenesis.

2. Geological Setting

Dongying Sag is a subtectonic unit in the southeastern of Jiyang Depression, Bohai Bay Basin (Figure 1) [35]. It can be further subdivided into six secondary tectonic zones, known as Northern Steep Slope Zone, Lijin Subsag, Minfeng Subsag, Central Anticline Zone, Niuzhuang Subsag, and Southern Gentle Slope Zone (Figure 1) [35].

There are mainly three sets of successions in the Dongying Sag, namely, Paleogene, Neogene, and Quaternary. The Paleogene mainly comprises Kongdian (Ek), Shahejie (Es), and Dongying (Ed) Formations; the Neogene Guantao (Ng) and Minghuazhen (Nm) Formations; and the Quaternary Pingyuan (Qp) Formation (Zhai and He 1993) [36]. There is a main regional unconformity serving as the boundary between Ed and Ng Formations (Figure 2) (Zhai and He 1993) [35, 36]. Shahejie Formation can be subdivided into four members, namely, Es4, Es3, Es2, and Es1, from base to top [36]. This study focused on the Es4 and Es3 members.

Es4 consists of gray and dark-gray mudstones, gypsum and halite, interbedded nearshore subaqueous fan sandstones, and sublacustrine fan sandstones deposited in semi-deep and deep lacustrine environments, which are mainly located in the Northern Steep Slope Zone. The lower submember of Es3 (Es3³) was deposited in semideep and deep lacustrine environments, dominated by lacustrine oil shales, dark-gray mudstones, calcareous mudstones, and subaqueous (sublacustrine) fan sandstones in terms of lithology. The middle submember of Es3 (Es3²) consists of gray to dark-gray mudstones, calcareous mudstones, subaqueous (sublacustrine) fan sandstones, and delta sandstones deposited in semideep and deep lacustrine environments. The upper submember of Es3 (Es3¹) is dominated by deltaic sandstones.

3. Samples and Methods

A total of 1472 thin sections were collected, which were prepared from the Es core samples in the Dongying Sag by Geological Scientific Research Institute of China Sinopec Shengli Oilfield Company, where 1423 data about Es formation water chemical composition were also collected.

A total of 300 blue epoxy resin-impregnated thin sections were prepared for diagenesis analysis using the Es drill cores of 46 wells in Dongying Sag. This study focused on the type, occurrence, content, and contact metasomatic relationship of diagenetic products. With at least 300 points, estimations of component contents by point accounting can be more

reliable with a standard deviation less than 5.5%. 40 out of 300 samples were implemented with EBSD, SEM, and EDX analysis in order to investigate chemical characteristics of authigenic kaolinites and carbon cements. 34 out of 300 samples showed only one type of carbon cements, and they were selected for analysis of $\delta^{13}\text{C}_{\text{V-PDB}}/\text{‰}$ and $\delta^{18}\text{O}_{\text{V-PDB}}/\text{‰}$. The collection of carbon cements from these samples was taken at the Institute of Mineral Resources and Regional Geology of Hebei Province, while the measurement of $\delta^{13}\text{C}_{\text{V-PDB}}/\text{‰}$ and $\delta^{18}\text{O}_{\text{V-PDB}}/\text{‰}$ was completed using Gas Isotope Ratio Mass Spectrometry (MAT 253) at Chinese Academy of Geological Sciences. Fluid inclusion analysis was carried out on 14 samples, which were prepared as doubly polished sections with approximate thicknesses of 100 μm for fluid inclusion petrographic analysis and thermometric measurement. Microthermometry of aqueous inclusions was conducted using calibrated Linkam-350, during which the homogenization temperature (Th) was obtained by cycling and Th measurements were completed with a heating rate of 10°C/min when the temperature was lower than 70°C, and 5°C/min when the temperature exceeded 70°C. The precision of measured Th was within $\pm 1^\circ\text{C}$. A total of 592 data concerning bulk rock and clay mineral analyses were collected from Geological Scientific Research Institute of China Sinopec Shengli Oilfield Company, which were mainly to identify the distribution of contents of different clay minerals.

4. Results

4.1. Detrital Composition of Es Sandstones. Es sandstones in Dongying Sag are medium to fine grained arkoses and lithic arkoses (av. $\text{Q}_{47.4}\text{F}_{33.8}\text{L}_{18.7}$; Figure 3). They are poorly to moderately sorted and subangular, with the matrix content of 1.7–13.6%. It was revealed by thin section observation that the quartz grains in those sandstones (av. 39.8%) were mainly monocrystalline quartz (av. 39.0%) and, less commonly, polycrystalline quartz (av. 0.8%). Es3 sandstones had the highest content of quartz grains (av. 43.2%), while Es1 sandstones had the lowest (av. 39.5%). The feldspar particles in those sandstones (av. 15.1%) were mainly K-feldspar (av. 9.0%) and, less commonly, plagioclase (av. 6.1%). Es1 sandstones had the highest content of feldspar particles (av. 20.2%), while Es3 sandstones had the lowest (av. 12.2%). The content range of lithic fragments in those sandstones was relatively large, 1.7–53.2%. Lithic fragments in Es sandstones were all dominated by metamorphic lithic fragments. The difference is that the amount of igneous and sedimentary lithic fragments was relatively higher in the Es1 and Es2 sandstones, but relatively lower in Es3 and Es4 sandstones (Table 1).

During the burial process, the interaction between pore fluids and grains in sandstones could result in the changes of type and content of clastic particles. In the sandstones with depth less than 3200 m, the content of quartz particles increased significantly, and the content of feldspar grains decreased dramatically, with the increase of depth. However, such trend was opposite in sandstones with depth more than 3200 m. Moreover, it was found that the content of lithic fragments kept relatively unchanged until the depth reached 2800 m, after which it sharply decreased with depth

TABLE 1: Modal composition (maximum, minimum, and average) of 1472 samples from the Es sandstones of Dongying Sag.

	Es1			Es2			Es3			Es4			Es
	Minimum (%)	Maximum (%)	Mean (%)	Minimum (%)	Maximum (%)	Mean (%)	Minimum (%)	Maximum (%)	Mean (%)	Minimum (%)	Maximum (%)	Mean (%)	Mean (%)
<i>Detrital composition</i>													
Monocrystalline quartz	26.2	59.0	38.5	16.0	61.8	39.4	24.0	69.6	42.5	21.8	43.4	39.5	39.0
Polycrystalline quartz	0.8	1.2	1.0	0.2	1.9	0.9	0.2	3.6	0.7	0.4	0.9	0.6	0.8
K-feldspar	1.7	26.5	10.4	1.0	25.9	10.5	1.0	16.5	7.4	5.8	16.4	7.7	9.0
Plagioclase	1.5	19.2	9.3	1.2	18.5	4.3	1.0	19.8	4.8	4.3	17.4	5.0	6.1
Volcanic lithic fragments	0.4	9.3	2.9	0.2	29.6	3.3	0.2	12.9	1.2	0.4	7.2	3.1	2.6
Metamorphic lithic fragments	1.4	15.0	3.3	0.7	38.5	4.1	0.7	12.5	8.1	2.6	27.7	4.4	5.5
Sedimentary lithic fragments	0.2	3.5	1.7	0.3	23.3	2.2	0.2	23.4	2.2	0.4	3.8	1.6	1.9
Mica	0.1	0.3	0.5	0.4	0.9	0.5	0.3	2.4	0.7	0.0	0.0	0.0	0.4
Charcoal debris	0.2	0.9	0.5	0.3	4.6	1.1	0.3	6.5	1.5	0.4	1.5	0.5	0.9
Clay matrix	0.9	11.9	3.6	0.2	12.8	3.4	0.4	9.6	2.2	0.7	12.6	5.0	3.3
Micritic matrix	0.9	6.9	2.1	0.4	4.5	1.2	0.2	3.3	1.4	0.0	0.0	0.0	1.4
<i>Diagenetic minerals</i>													
Calcites	2.8	33.0	5.2	0.2	28.6	3.1	0.2	28.3	1.7	0.5	3.1	1.2	2.8
Dolomites	0.0	0.7	0.4	1.5	18.5	2.2	0.3	22.8	1.6	0.4	7.5	3.4	1.9
Ferrocacites	0.0	1.8	0.9	0.0	27.5	3.1	0.3	31.3	6.2	0.7	8.4	2.6	3.2
Ankerites	0.0	3.3	1.6	0.0	22.6	3.7	0.4	25.9	4.0	0.5	9.8	6.2	3.9
Authigenic quartz	0.0	0.0	0.0	0.4	1.8	0.8	0.2	3.4	1.1	0.4	3.5	1.0	0.7
pyrites	0.0	2.6	1.8	0.0	5.0	1.3	0.4	4.5	1.3	0.2	6.7	2.5	1.7
Authigenic kaolinites	0.0	0.0	0.0	0.0	3.9	2.7	0.4	9.4	3.2	0.0	1.6	0.3	1.6
Fe-oxide	0.0	0.0	0.0	0.0	1.5	0.2	0.4	5.8	0.7	0.7	4.2	2.6	0.9
Authigenic chlorites	0.0	1.3	0.7	0.0	2.3	0.4	0.0	4.6	1.3	0.6	5.7	3.2	1.4
Authigenic illites	0.0	0.0	0.0	0.0	0.0	0.0	0.0	3.2	1.1	0.1	4.3	2.5	0.9
Siderite	0.0	0.0	0.0	0.4	6.4	1.1	0.0	2.1	0.2	0.0	0.0	0.0	0.3
<i>Porosity</i>													
Primary pore	15.0	20.5	13.9	5.4	17.3	7.2	0.0	7.3	0.5	0.0	6.2	0.7	5.6
Feldspars													
dissolution pores	0.0	2.6	1.2	0.0	7.5	3.6	0.0	5.3	1.8	0.0	4.2	2.0	2.2
Carbonate													
dissolution pores	0.0	0.0	0.0	0.0	2.1	0.4	0.0	10.5	2.2	0.0	8.9	4.3	1.7
Fractures	0.0	0.9	0.5	0.0	1.2	0.7	0.0	1.3	0.5	0.0	1.7	0.2	0.5

FIGURE 1: Location map and cross section of the study area. (a) Location map of the study area showing the subtectonic units of the Bohai Bay Basin, namely, Jizhong Depression (I), Huanghua Depression (II), Jiyang Depression (III), Bozhong Depression (IV), and Liaohe Depression (V). (b) Structural map of the Dongying Sag. (c) N-S cross section (P'-P) of the Dongying Sag showing the various tectonic-structural zones and key stratigraphic intervals [35].

(Figure 4). In addition, in the depth less than 2800 m, the proportion of different lithic fragments in total lithic fragments was relatively constant, which, however, changed when the depth reached 2800 m. To be specific, the proportion of igneous lithic fragments increased with depth, while

that of metamorphic lithic fragments decreased (Figure 4). Similarly, the proportion of anorthose grains in total feldspar grains declined with depth, while that of K-feldspar grains increased, when the depth was less than 2800 m (Figure 4). However, in sandstones with depth from 2800 m to 3200 m,

FIGURE 2: Generalized Cenozoic-Quaternary stratigraphy of the Dongying Sag (modified from [35]).

the trend was opposite, which meant increasing anorthose grains and decreasing K-feldspar grains with depth. As for the sandstones buried deeper than 3200 m, contents of anorthose and K-feldspar rarely changed (Figure 4).

4.2. Chemical Characteristics of Formation Water in Dongying Sag. The major elements of formation water were found to change regularly with depth in Dongying Sag. The contents of Na⁺ and K⁺ increased in the depth ranges of 2200 m–2500 m and 2800 m–3400 m, respectively, reaching the maximum at the depth of 3400 m and then slightly declining. The content of Cl[−] increased with depth from 2200 m, achieving the maximum and remaining stable after the depth exceeded 2500 m. The content of Ca²⁺ greatly increased with depth

in the range of 2200 m–3200 m, reaching the maximum at 3200 m and then dropping greatly with depth. The content of HCO₃[−] increased in the range of 2500 m–3000 m, achieving the maximum at 3000 m and then decreasing greatly with depth. The content of CO₃^{2−} increased with depth after the depth exceeded 2800 m (Figure 5).

4.3. Morphological, Geochemical, and Distribution Features of Diagenetic Products in Es Sandstones. Pore fluids in Es sandstones experienced multiphase material exchange and migration, leading to the formation and evolution of diagenetic products. These diagenetic products mainly included carbonate cements; quartz cements; aluminosilicate minerals such as kaolinite, illite, and chlorite; opaque minerals such

FIGURE 3: Detrital composition of sandstone samples of 1472 samples from the Es sandstones in Dongying Sag.

as pyrite; dissolution pores of unstable particles (feldspar and lithic fragments) and carbonate cements. The contents of different diagenetic products varied greatly in the sandstones of different members of Es. The contents of carbonate cements were relatively higher in Es3 and Es4 sandstones (av. 13.5% and av. 13.4%, resp.), while that in Es1 sandstones was relatively lower (av. 8.1%). The carbonate cements in Es1 sandstones were mainly calcite; those in Es2 sandstones calcite, dolomite, ferrocalcite, and ankerite; those in Es3 sandstones mainly ferrocalcite and ankerite; and those in Es4 sandstones mainly dolomite and ankerite. Quartz cements were mainly observed in sandstones of Es2, Es3, and Es4, with rare observations of them in Es1 sandstones. Authigenic kaolinites were mainly found in Es2 and Es3 sandstones, while few of them were observed in sandstones of Es1 and Es4. Feldspar dissolution pores were predominantly in sandstones of Es2, Es3, and Es4, and they were most developed in Es3 sandstones. Es3 and Es4 sandstones were main hosts to carbonate cement dissolution pores, and Es4 sandstones had the largest amount of carbonate cement dissolution pores (Table 1).

4.3.1. Distribution and Geochemical Features of Carbonate Cements with Different Morphologies. There were mainly four stages of carbonate cements with different morphologies in the study area. Carbonate cements of the first stage (Cc1) were generally isopachous on the surface of particles (Figures 6(a) and 6(b)), occurring in sandstones with depth ranging from 1700 m to 3600 m (Figure 7). They were only observed

in a few sandstone samples. Carbonate cements of the second stage (Cc2) were mainly medium-coarse crystalline calcite, filling the primary pores which were not obviously affected by compaction. This type of carbonate cements was generally on the outer side of Cc1 (Figures 6(a), 6(b), and 6(c)), and they usually occurred in sandstones with depth ranging from 1700 m to 3600 m, concentrated in the depth range of 1700–2800 m. From 2800 m to 3600 m, Cc2 was obviously dissolved (Figure 7). Carbonate cements of the third stage (Cc3) filled in residual primary pores after compaction as well as feldspar dissolution pores (Figures 6(e) and 6(f)). They generally occurred in the sandstones with depth ranging from 2000 m to 3600 m, concentrated in the range of 2100 m to 2700 m. The content of Cc3 decreased greatly with depth once the depth reached 2700 m (Figure 7). Carbonate cements of the fourth stage (Cc4) were euhedral fine crystalline ferrocalcites and ankerites, lying on the outer part of kaolinization feldspar or secondary pores dissolved by Cc3 (Figures 6(g) and 6(h)). They were mainly found in the sandstones with the depth over 3100 m, and their content increased greatly with depth (Figure 7).

Cc1 was mainly micritic high-Mg calcite (CaCO_3 71–79%; MgCO_3 15–23%; FeCO_3 2–5%; MnCO_3 0–2%) (Figure 8). It was difficult to obtain isotope data from Cc1, due to its low content. Cc2 was mainly calcite (CaCO_3 91–100%; MgCO_3 0–5%; FeCO_3 0–5%; MnCO_3 0–2%) (Figure 8), with $\delta^{13}\text{C}_{\text{V-PDB}}/\text{‰}$ values from +1.60‰ to +3.50‰ (av. 2.76‰) and $\delta^{18}\text{O}_{\text{V-PDB}}/\text{‰}$ values from –12.40‰ to –9.10‰ (av. –10.33‰) (Figure 9). The chemical composition of Cc3

FIGURE 4: The content changes of different grains in sandstones of Es in Dongying Sag.

FIGURE 5: Changes of chemical characteristics of formation water with depth in Dongying Sag.

was complicated ($CaCO_3$ 43–97%; $MgCO_3$ 0–54%; $FeCO_3$ 1–11%; $MnCO_3$ 0–4%) (Figure 8), and it had a relatively wide range of $\delta^{13}C_{V-PDB}/\text{‰}$ values from -6.60‰ to $+4.30\text{‰}$ (av. 1.03‰) and $\delta^{18}O_{V-PDB}/\text{‰}$ values from -13.90‰ to -5.10‰ (av. -11.00‰) (Figure 9). Cc4 had higher contents of Fe and Mn ($CaCO_3$ 38–83%; $MgCO_3$ 3–53%; $FeCO_3$ 6–14%; $MnCO_3$ 0–7%) (Figure 8), with the $\delta^{13}C_{V-PDB}/\text{‰}$ values from

-6.40‰ to -3.30‰ (av. -4.85‰), and $\delta^{18}O_{V-PDB}/\text{‰}$ values from -15.90‰ to -13.50‰ (av. -14.70‰) (Figure 9).

4.3.2. Distribution of Quartz Cementation with Different Morphologies and Fluid Inclusions. Three types of quartz cementation could be identified according to the morphology, respectively, in forms of quartz overgrowth, micro- to

FIGURE 6: Morphological features of diagenetic products in Es of Dongying Sag. ((a) Well Shi127 2180.8 m, micritic high-Mg calcite (Cc1) and medium-coarse calcites (Cc2); (b) Well Shi127 2180.8 m, local amplification of (a), Cc1, Cc2, and Py1; (c) Well Xin 154 2960.50 m, Cc2 partly filled in primary pores and the outer part was filled by K1; (d) Well Xin 154, 2960.50 m, local amplification of (c); (e) Well Niu110, 3004.80, dissolution of Cc3 and fillings in the outer of Q1; (g) Well FS1, 4322.40 m, Cc4 occurred in the outer of kaolinization feldspar (K2); (h) Well FS1, 4322.40 m, local amplification of (g)).

FIGURE 7: The distribution of carbonate cements with different morphological features in sandstone of Es in Dongying Sag.

FIGURE 8: Geochemical characteristics of carbonate cements with different morphologies in Es of Dongying Sag.

mega- crystalline pore-filling quartz cements, and fracture-filling quartz cements (Figure 10). The first stage of quartz cements (Q1) was mainly in forms of quartz overgrowth, and the outer part was filled by Cc3 (Figures 6(e), and 6(f),

13(a), 13(b), and 13(c)). Q1 had a depth ranging from 2000 m to 3600 m, mainly concentrated in 2500–3600 m (Figure 11). The second stage of authigenic quartz (Q2) was mainly euhedral quartz and fracture-filling quartz, filled in the outer

FIGURE 9: Isotope characteristics of carbonate cements with different morphologies in Es sandstones, Dongying Sag [37–46].

part or cutting Q1 (Figures 10(a) and 10(b)). Q2 was in a depth ranging from 2500 m to 3600 m and was mainly concentrated in 2900 m–3600 m (Figure 11).

The homogenization temperature of inclusions in Q1 ranged from 70°C to 115°C and that in Q2 from 110°C to 130°C (Figures 10(a), 10(b), and 12).

4.3.3. Distribution and Geochemical Features of Authigenic Kaolinites with Different Morphologies. The morphological features of authigenic kaolinites were comprehensively studied by thin section observation and SEM analysis. The authigenic kaolinites in the study area could be classified into two categories based on their morphologies. The first kind of authigenic kaolinite (K1) was featured by predominant scaly-shape under the microscope (Figures 6(c), 13(a), and 13(c)) as well as single crystals characterized by closely packed, thin complete pseudo-hexagonal flakes under SEM. The wormlike or book-like aggregation of K1 (Figures 6(d), 13(b), and 13(d)) was mainly in feldspar dissolution pores and residual primary pores which had been partially filled by Cc2 (Figure 6(c)). K1 intergrew with quartz overgrowth (Q1) (Figure 13(b)), and it could be replaced by illites (Figure 13(g)). K1 had a wide range of distribution from 1700 m to 3600 m and was mainly concentrated in the range of 2600 m to 3200 m (Figure 14). Under microscopes, the second kind of authigenic kaolinite (K2) was mainly distributed on the surface of feldspar particles, showing disordered scales, and the aggregate of K2 presented in the shape of feldspar particles (Figures 6(g) and 13(e)). Under SEM, single crystals of K2 were flake-shaped, thin, and loosely arranged. These crystals had curved edges and imperfect forms (Figure 13(f)). K2 was mainly in dissolution pores within Cc3, with the outer side filled by Cc4 (Figures

6(g) and 6(h)). Part of K2 intergrew with authigenic quartz (Q2). K2 was mainly in the range of 2600 m to 3600 m and was mainly concentrated in the range of 2900–3200 m (Figure 14).

K1 mainly contained three elements, namely, Al, Si, and O, while K2 had additional trace elements such as K (av. 1.01%) and Fe (av. 1.89%) (Table 2).

4.3.4. Morphological and Distribution Characteristics of Other Water-Rock Reaction Products. In the sandstones with depth less than 3200 m, contents of illite (less than 5%) and chlorite (less than 2%) were relatively low and stable, while that of kaolinite increased significantly with depth (Figure 15). However, when the burial depth reached 3200 m, the contents of illite and chlorite began to increase with depth, while that of kaolinites dropped (Figure 15), which was attributed to the transformation of kaolinites into illite and chlorite as observed under SEM (Figures 6(h), 13(g)).

There were mainly two kinds of authigenic pyrites in Es sandstones, Dongying Sag. The first kind of pyrites (Py1) occurred as single particles in the inner side of Cc1 (Figure 6(b)), while the other kind (Py2) occurred as framboids around Cc4 (Figure 13(h)).

5. Discussion

5.1. Formation Timing and Diagenetic Sequence of Major Diagenetic Products. The $\delta^{18}\text{O}_{\text{smow}}/\text{‰}$ value of carbonate cements was controlled by formation temperatures and $\delta^{18}\text{O}_{\text{smow}}/\text{‰}$ values of pore fluids. It was necessary to firstly determine $\delta^{18}\text{O}_{\text{smow}}/\text{‰}$ values of pore when using $\delta^{18}\text{O}_{\text{smow}}/\text{‰}$ value of carbonate cements to calculate the formation

FIGURE 10: The inclusion characteristics of authigenic quartz in Dongying Sag. ((a) Well Y67, 2960.50 m; (b) Well S126, 3450.4 m).

TABLE 2: Element component characteristics of authigenic kaolinites in Es of Dongying Sag.

Well ID	Depth (m)	Type	Element content percentage (mol%)				
			O	Al	Si	K	Fe
S 126	3386.40	K1	60.15	18.38	21.47	—	—
S 126	3386.40	K1	60.37	18.22	21.41	—	—
S 126	3428.60	K1	52.99	21.16	25.85	—	—
S 126	3428.60	K1	56.62	19.51	23.87	—	—
X 154	2935.90	K1	58.34	18.87	22.79	—	—
N 105	3096.20	K1	56.61	20.24	23.15	—	—
H 159	2960.30	K1	68.16	13.79	18.05	—	—
S 126	3513.1	K1	55.02	20.14	24.84	—	—
S 126	3450.40	K1	65.32	16.02	18.66	—	—
S 128	3452.40	K1	51.47	21.51	27.02	—	—
S 126	3450.40	K2	48.13	19.94	27.70	2.23	2.00
S 126	3450.40	K2	50.80	19.61	28.28	0.52	0.78
S 126	3450.40	K2	50.55	13.47	31.95	1.20	2.84
N 105	3096.20	K2	56.90	15.58	25.13	0.76	1.63
N 105	3096.20	K2	51.08	20.97	27.19	0.76	—
N 105	3096.20	K2	49.41	22.03	28.07	0.49	—
S 126	3513.1	K2	55.00	20.44	23.02	0.68	0.86
S 126	3513.1	K2	53.64	18.30	25.27	1.63	1.16
S 127	3451.40	K2	48.95	20.63	28.01	0.95	1.46
S 130	3454.40	K2	47.52	21.64	27.50	—	3.34
S 131	3455.40	K2	52.62	18.50	25.05	0.87	2.96

temperature [37, 48]. During diagenetic process, material exchanges between particles and pore fluids (mainly feldspar dissolution) led the $\delta^{18}\text{O}_{\text{smow}}/\text{‰}$ value of pore fluids to be heavier [35, 39, 43]. The $\delta^{18}\text{O}_{\text{smow}}/\text{‰}$ value of pore water at eodiagenetic stage was about -4.8‰ , which became heavier, reaching about -3‰ due to significant feldspar dissolution [35, 49]. In other words, Cc1 and Cc2, which were clearly anterior to feldspar dissolution, precipitated from pore fluids with $\delta^{18}\text{O}_{\text{smow}}/\text{‰}$ value of -4.8‰ (Figures 6(a), 6(b), and 6(c)), while Cc3 and Cc4, which were clearly posterior to

feldspar dissolution, precipitated from pore fluids with $\delta^{18}\text{O}_{\text{smow}}/\text{‰}$ value of -3‰ (Figures 6(e), 6(f), 6(g), and 6(h)). The chemical composition of Cc1 was similar to micritic high-Mg calcite formed during syndiagenetic stage in shales (Figure 8). Thus, it could be deduced that Cc1 should be formed during syndiagenetic stage as well. The $\delta^{18}\text{O}_{\text{V-PDB}}/\text{‰}$ values of Cc2 ranged from -12.40‰ to -8.20‰ , indicating the formation temperature range of Cc2 to be $33.2\text{--}58.1^\circ\text{C}$. The $\delta^{18}\text{O}_{\text{V-PDB}}/\text{‰}$ value of Cc3 ranged from -13.90‰ to -5.10‰ , suggesting the formation temperature range of Cc3

FIGURE 11: The distribution of authigenic quartz with different morphological features in sandstone of Es in Dongying Sag.

FIGURE 12: The homogenization temperature of inclusion of authigenic quartz with different morphological features in sandstones of Es, Dongying Sag.

to be 50.0–115.2°C. Similarly, the $\delta^{18}\text{O}_{\text{V-PDB}}/\text{‰}$ of Cc4 ranged from –15.90‰ to –13.50‰, implying the formation temperature range of Cc3 to be 130–170°C [37, 48].

The homogenization temperatures of brine inclusions in Q1 and Q2, respectively, ranged from 70°C to 115°C and from 110°C to 130°C (Figures 10(a), 10(b), and 12).

K1, which was posterior to cementation of Cc1 and Cc2 and prior to Cc3, generally presented as intergrowth of Q1 (Figures 6(c), 6(h), and 13(b)), indicating the formation temperature range of K1 to be 70–115°C. K2, which was posterior to cementation of Cc3 and anterior to Cc4, generally occurred as intergrowth of K2, implying the formation temperature of K1 to range from 115°C to 130°C.

Chlorite and illite were synchronous with or posterior to Cc4 and were concentrated in depth more than 3200 m

(>140°C), suggesting that they were the latest diagenetic products (Figure 6(h), and 15).

Pyl was generally associated with Cc1 (Figure 6(b)) and Py2 with Cc4 (Figure 13(h)).

Finally, the diagenetic sequence of sandstones in Es, Dongying Sag, was concluded as shown in Figure 16.

5.2. Material Sources of Main Diagenetic Products

5.2.1. Material Sources of Carbonate Cements. Cc1 could be directly precipitated out from sedimentary waters during syndiagenetic stage [51]. The $\delta^{13}\text{C}_{\text{PDB}}/\text{‰}$ and $\delta^{18}\text{O}_{\text{PDB}}/\text{‰}$ values of Cc2 in sandstones ranged from 1.60‰ to 3.50‰ and from –12.4‰ to –8.2‰, respectively, indicating Cc2 to be typical lacustrine carbonate cements (Figure 9). Cc2 mainly occurred in sandstones which were proximal to sand-shale interfaces [52–55]. Formation temperatures of Cc2 were within the range of 33.2–58.1°C, which corresponded to the depth range of 500–1000 m [50]. In this depth range, the porosity of mudstones declined from 60% to 10–20% because of compaction, leading to discharge of a large amount of sedimentary water into sandstones [13, 53, 55–58]. Ca^{2+} and CO_3^{2-} were rich in those fluids, and they were main material sources for Cc2.

The $\delta^{13}\text{C}_{\text{PDB}}/\text{‰}$ and $\delta^{18}\text{O}_{\text{PDB}}/\text{‰}$ values of Cc3 in sandstones mainly ranged from –6.6‰ to 4.3‰ and from –13.9‰ to –5.1‰, respectively, which indicated the influence of organic carbon on part of Cc3 (Figure 9). The chemical composition of Cc3 was complex (Figure 8). Vertically, Cc3 was mainly concentrated in transitional areas of normal-pressure and overpressure zones (Figure 7; [59]). Laterally, distribution of Cc3 was controlled by main faults [59]. All of these proved that hydrothermal fluids that flowed through the faults provided part of the material sources for Cc3. Formation temperatures of Cc3 ranged from 50.0°C to 115.2°C, in the range of which carboxylic acids were formed and expelled from organic matters into mudstones [13, 60]. The presence of carboxylic acids led to dissolution of plagioclases (mainly Ca-feldspars and Na-feldspars) and also caused the entrance of Ca^{2+} and Na^{+} into pore fluids (Figures 4, 6(f), and 5). During this time, no obvious dissolution of Cc1 and Cc2 occurred (Figure 6(c), Figure 7; [61]). The Ca^{2+} , Mg^{2+} , and CO_3^{2-} (partly influenced by organic carbon) in hydrothermal fluids and Ca^{2+} (dissolved by Ca-feldspar) and CO_3^{2-} (sedimentary carbon) in formation water were material sources for Cc3 [59, 62, 63].

The $\delta^{13}\text{C}_{\text{PDB}}/\text{‰}$ and $\delta^{18}\text{O}_{\text{PDB}}/\text{‰}$ values of Cc4 in sandstones ranged from –6.4‰ to –3.3‰ and from –15.9‰ to –13.5‰, respectively, indicating the significant influence of organic carbon on Cc4 (Figure 9). Cc4 was mainly concentrated in sandstones with depth over 3200 m (Figure 7). At this depth (corresponding temperature > 120°C), cracking of carboxylic acids and organic matters resulted in a large amount of CO_2 , which was transformed to CO_3^{2-} in the following diagenetic processes [13, 27, 35, 50, 54, 60]. Carbonate cements (mainly Cc2 and Cc3) were dissolved obviously, leading to the entrance of Ca^{2+} and CO_3^{2-} into pore fluids (Figures 6(e), and 7; [64]). The dissolution of metamorphic

FIGURE 13: Morphological features of diagenetic products in Es of Dongying Sag. ((a) Well He 142, 3064.83 m, wormlike authigenic kaolinites (K1) intergrew with quartz overgrowths; (b) Well He 142, 3064.83 m, local amplification of (a), K1 intergrew with quartz overgrowths; (c) Well Xin 154, 2938.56 m, K1 filled in secondary pores; (d) Well Xin 154, 2938.56 m, local amplification of (c), wormlike authigenic kaolinite (K1); (e) Well N105, 3096.2 m, disordered authigenic kaolinite distributed on the surface of K-feldspars (K2); (f) Well N105, 3096.2 m, local amplification of (e); (g) Well XXI161, 3272.5 m, illitization of K1 and K2; (h) Well S106, 3398.70 m, Cc4 and Py2).

FIGURE 14: The distribution of authigenic kaolinites with different morphologies in Es sandstone of Dongying Sag [47].

lithic fragments promoted the entrance of Fe^{2+} and Mg^{2+} into pore fluids (Figure 4). All of these materials were the sources for precipitation of Cc4.

5.2.2. Material Sources of Authigenic Quartz. Precipitation of authigenic quartz occurred in the diagenesis process (Figure 16) as the concentration of SiO_2 (aq) (<100 ppm) in lake waters was too low for authigenic quartz [11]. There were no external sources of free SiO_2 for sandstones of Es in Dongying Sag [35]. However, quartz dissolution at grain contacts and dissolution or transformation of feldspars in sandstones could be possible internal source for authigenic quartz. Contact relationships of Es sandstones were mainly point contact, with a small proportion of line contact (Figures 6(a), 6(c), 13(a), and 13(c)). No much free SiO_2 (aq) was released during this process. Therefore, the depth distribution range of authigenic quartz was identical to that of feldspar (Figures 4 and 11). In the thin sections and SEM, dissolution and transformation of feldspars associated with authigenic quartz could be identified (Figure 13(b)). Therefore, the most likely source of authigenic quartz might be the internal dissolution and transformation of feldspars.

5.2.3. Material Sources of Authigenic Kaolinites. The concentrations of SiO_2 (aq) (<100 ppm) and Al^{3+} (<10 ppm) were too low to be effective material source for authigenic kaolinites [11]. However, there were a lot of authigenic kaolinites in the sandstone reservoirs of Es in Dongying Sag. Vertically, the content of authigenic kaolinites was negatively correlated with feldspar content (Figures 4 and 14; [35, 47, 65]). In other words, the dissolution of aluminosilicate minerals (mainly feldspars) was important material source for authigenic kaolinites [9, 66–71].

K1 was featured by perfect crystal forms and exclusive Al, Si, and O ions, indicating those authigenic kaolinites to be

precipitated directly from pore fluids [72, 73]. High content of K1 often appeared in samples with no or little feldspar dissolution, while low content of K1 was common in samples with a large amount of feldspar dissolution [74], which indicated the significant amount of free Si^{4+} and Al^{3+} which are released by dissolution of feldspar and then migration in pore fluids during the formation of K1. In the deep burial environment, pore fluids flowed slowly, dissolving only a small amount of free Si^{4+} and Al^{3+} . Therefore, it was hard for silicon and aluminum to migrate in the form of Si^{4+} and Al^{3+} [75–77]. Considering the formation temperature (60–115°C) of K1, which was suitable for formation and preservation of carboxylic acids, it was suggested that complex reaction between carboxylic acids and cations of Si^{4+} and Al^{3+} could induce the dissolution of feldspars [27, 35, 78]. The Si^{4+} and Al^{3+} migrated over long distances in the form of clathrates and precipitated as authigenic kaolinite (K1) in the proper geological environment.

K2 was featured by imperfect crystal forms and more other ions (like Fe and K) besides Al, Si, and O, which indicated these authigenic kaolinites to be products of feldspar transformation [72, 73]. Pore fluids rich in CO_2 led to the occurrence of a large amount of H^+ and HCO_3^- in pore fluids at the depth of 2800–3200 m (Figure 5). Under this situation, K-feldspar particles were totally or partly transformed into K2 and Q2 [5, 61].

5.2.4. Material Sources of Other Water-Rock Reaction Products. In the depth range of 2800–3200 m, kaolinization of K-feldspars and dissolution of metamorphic lithic fragments led to concentration of K^+ , Fe^{2+} , and Mg^{2+} in pore fluids. At the same time, a large amount of H^+ was consumed. All of these led authigenic kaolinites (K1 and K2) to be unstable [56, 79]. Once the depth was above 3200 m (130°C), K1 and K2 were partly or totally transformed into illites and chlorites (Figures 6(h), 13(g), and 15).

5.3. Material Exchanges between Pore Fluids and Rocks during Diagenetic Processes. The material exchange between pore fluids and rocks was mainly controlled by geochemical and physical properties of pore fluids. During diagenetic processes, fluids, which were recharged by mudstones and flowed along faults, could lead to obvious changes of properties of pore fluids. Based on analyses of changes of detrital composition, geochemical features of pore fluids, and material sources of diagenetic products in Es of Dongying Sag, this study divided the material exchanges during diagenetic processes in rift basins into five stages.

Stage 1. Certain amounts of Ca^{2+} , Mg^{2+} , Fe^{3+} , CO_3^{2-} , and SO_4^{2-} were existent in lake waters of the Paleogene in Dongying Sag [51, 80, 81]. During syngenetic stage, evaporation of lake water led to concentration of ions, and they precipitated as a small amount of micritic high-Mg calcite [51, 54, 81–83]. At the same time, activities of sulfate-reducing bacteria led to the change of Fe^{3+} and SO_4^{2-} to Fe^{2+} and S^{2-} , respectively. Part of Fe^{2+} entered into crystal lattices of Cc1 as

FIGURE 15: The content changes of clay minerals in Es sandstones from Dongying Sag.

FIGURE 16: The diagenetic sequence of Es in Dongying Sag (paleogeothermal data was based on [50]; burial history was based on [47]).

heteroatom, while the remaining part was combined with S^{2-} , precipitating as Py1 associated with Cc1. In this stage, mainly Mg^{2+} , Ca^{2+} , CO_3^{2-} , Fe^{2+} , and SO_4^{2-} , which originated from lake waters, entered sandstones in the forms of Cc1 and Py1.

Stage 2. In the depth range of 500–1000 m (paleotemperature of 30–50°C), concentrated compaction waters rich in Ca^{2+} and CO_3^{2-} entered into sandstones from mudstones, resulting in the formation of Cc2 in sandstones close to the sandstone-mudstone interfaces. During this stage, Ca^{2+} and CO_3^{2-} from compaction waters entered into sandstones and precipitated as Cc2.

Stage 3. In the depth range of 1250–2800 m (paleotemperature of 60–120°C), waters rich in carboxylic acids enter into sandstones from mudstones, leading to dissolution of plagioclases. In this process, Na^+ and Ca^{2+} entered into pore fluids and were well preserved. Si^{4+} and Al^{3+} were complexed with carboxylic acids, and these complexes migrated in pores and then precipitated as K1 and Q1 in the proper geological conditions. In this stage, activation of faults could cause the blend of hydrothermal fluids (upwelling through faults, rich in Mg^{2+} , Ca^{2+} , and partly organic source CO_3^{2-}) and formation waters (in place, rich in Ca^{2+}), leading to the precipitation of Cc3.

Stage 4. In the depth range of 2800–3200 m (paleotemperature of 120–140°C), CO_2 with organic sources, formed by cracking of organic matters and carboxylic acids, entered the pore fluids and was preserved as H_2CO_3 (HCO_3^-). The presence of organic source H_2CO_3 (HCO_3^-) led to the transformation of K-feldspars and the dissolution of Cc2, Cc3, and metamorphic lithic fragments. During transformation of K-feldspars, a large amount of K^+ entered into pore fluids and was well preserved. The dissolution of Cc2 and Cc3 led to the entrance of a large amount of sedimentary source Ca^{2+} and CO_3^{2-} into pore fluids, which were then well preserved. The dissolution and transformation of metamorphic lithic fragments led to the entrance of Fe^{2+} and Mg^{2+} into pore fluids and subsequently good preservation (Figure 5). The transformation of K-feldspars led to the formation of K2 and Q2. In this process, K^+ , Ca^{2+} , CO_3^{2-} , Mg^{2+} , Fe^{2+} , and Mn^{2+} entered pore fluids.

Stage 5. In the depth above 3200 m (paleotemperature above 140°C), less or no formation of organic CO_2 resulted in the transformation of pore fluids into alkaline. In this depth range, the contents of Na^+ , K^+ , Ca^{2+} , and HCO_3^- significantly declined (Figure 5), which might be due to the precipitation of Cc4 as well as illitization and chloritization of K. During this process, because of declining of H^+ , HCO_3^{2-} transformed to CO_3^{2-} . This caused CO_3^{2-} (dissolution of Cc2 and Cc3, organic source CO_3^{2-}), Ca^{2+} , Mg^{2+} , Fe^{2+} , and Mn^{2+} to precipitate as Cc4. Concentration of K^+ and declining of H^+ contributed to the transformation of K1 and K2 into illites, while concentration of Mg^{2+} and Fe^{2+} as well as declining of H^+ led to the transformation of K1 and K2 into chlorites. In this process, CO_3^{2-} , Ca^{2+} , Mg^{2+} , Fe^{2+} , Mn^{2+} , and K^+ entered sandstones in the forms of Cc4, illites, and chlorites.

6. Conclusions

The material exchange of pore fluids in rift basins could be divided into five stages. The first stage was the near surface evaporation concentration stage, during which Ca^{2+} , Mg^{2+} , and CO_3^{2-} in lake waters precipitated as high-Mg calcites (Cc1), mainly caused by evaporation. The second stage was the shale compaction stage, during which Ca^{2+} and CO_3^{2-} from shale compaction waters precipitated as calcites (Cc2), mainly caused by compaction of shales. The third stage was the carboxylic acid dissolution stage, during which dissolution of plagioclases (by carboxylic acid) occurred. During this stage, Ca^{2+} and Na^+ entered into pore fluids as ions, while Si^{4+} and Al^{3+} entered into pore fluids and migrated as clathrates, ultimately precipitating as kaolinites (K1) and quartz overgrowth (Q1). Partly, the upwelling of hydrothermal fluids caused by active faults led to the precipitation of carbon cements (Cc3). Those processes were mainly caused by carboxylic acids and upwelling of hydrothermal fluids. The fourth stage was the organic CO_2 stage, which was featured by the kaolinization of K-feldspar, formation of organic CO_2 , and dissolution of metamorphic lithic fragments and carbon cements (mainly Cc2 and Cc3). During this stage, K^+ , Fe^{2+} , Mg^{2+} , Ca^{2+} , HCO_3^- , and CO_3^{2-} entered into pore fluids, driven by formation of organic CO_2 . The fifth stage was the alkaline fluid stage, which was characterized by the cementation of ferro-carbonates and ankerite as well as illitization or chloritization of kaolinites. During this stage, K^+ , Fe^{2+} , Mg^{2+} , Ca^{2+} , and CO_3^{2-} precipitated from pore fluids and entered into sandstones, caused by declining concentration of H^+ .

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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