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沁水盆地中南部太原组煤系页岩孔隙结构特征

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摘要:页岩孔隙结构及其控制因素的研究,对页岩气的勘探开发具有重要的指导意义。对沁水盆地太原组海陆过渡相煤系页岩进行了地球化学及低温气体吸附等试验,分析了过渡相页岩的地球化学特征、矿物组成特征和孔隙特征,在此基础上探讨了孔隙发育的控制因素。结果表明,沁水盆地太原组海陆过渡相煤系页岩有机碳质量分数为1.55%~5.21%,平均为3.53%,有机质类型主要为Ⅲ型,且多处于高-过成熟阶段。除少量的长石及碳酸盐矿物外,矿物质组成以质量分数占比55%~69%的黏土矿物和27%~40%的石英为主。黏土矿物中则以高岭石质量分数最高(为23%~65%,平均为48%),其次为伊蒙混层、伊利石和绿泥石。中孔对页岩孔隙体积的贡献最大(贡献率为35.6%~53.7%),其次为大孔(贡献率为25.7%~40.8%)。微孔对孔隙体积的贡献最小,但对表面积的贡献最大(贡献为72.2%~88.2%),而大孔对表面积的贡献几乎可以忽略,仅占总表面积1.5%~2.2%。有机碳含量对微孔及中孔的发育有一定正面影响,但对大孔的影响较小,热演化则不利于孔隙的发育。石英不利于海陆过渡相煤系页岩孔隙的发育,而黏矿物则对孔隙发育有促进作用,尤其是对中孔发育的影响尤为显著,其次是微孔,对大孔影响较弱。黏土矿物中,高岭石和绿泥石有利于页岩孔隙的发育,而伊利石和伊蒙混层则抑制了过渡相页岩孔隙的发育。

关键词:页岩气;孔隙结构;控制因素;海陆过渡相;沁水盆地

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Pore structure characteristics of Taiyuan Formation coal measures shale in south central Qinshui Basin

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Abstract:The study of the pore structure and its controlling factors of shale reservoir has important guiding significance for further exploration and exploitation of shale gas. Geochemical and low-temperature gas adsorption experiments were carried out on the transitional coal measures shale of Taiyuan Formation in Qinshui Basin. The geochemical characteristics, mineral matter composition characteristics and pore features were analyzed. On this basis, the controlling factors of pore development were discussed. The results show that the total organic carbon (TOC) content of the transitional shale of Taiyuan Formation ranges from 1.55% to 5.21%, with an average of 3.53%, the type of organic matter is mainly type III, and most of them are in a stage of highly to over mature stage. In addition to a small amount of feldspar and carbonate minerals, the two main components of this shale are 55%~69% of clay minerals and 27%~40% of quartz. Among the clay minerals, the content of kaolinite is the highest (ranging from 23% to 65%, with an average of 48%), followed by an illite mixed layer, illite and chlorite. Mesopores are the major contributor to pore volume (between 35.6% and 53.7%), followed by macropore (between 25.7% and 40.8%). The contribution of micropores to pore volume is the smallest, but the contribution of specific surface area is the lar-

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gest (between 72.2% and 88.2%), while the contribution of macropores to specific surface area is almost negligible, accounting for only 1.5~2.2% of the total specific surface area. The TOC content has a positive effect on the development of micropores and mesopores, but has little effect on macropores, and thermal evolution is not conducive to the development of pores. Quartz inhibit the development of pores, while the total amount of clay minerals is beneficial to the development of pores, especially to mesopore, followed by micropores and weak effects on macropores. In clay minerals, kaolinite and chlorite are beneficial to the development of shale pores, while the illite mixed layer and Illite inhibit the development of pores in the transitional shale.

Key words: shale gas; pore structure; controlling factor; marine-continental transitional; Qinshui Basin

0 引言

全球能源需求的增加及技术的进步使得页岩气这一非常规资源受到了关注^[1-2]。页岩气最初在北美实现了成功的商业开发,近年来,我国页岩气勘探和开发也取得了实质性进展^[3-4]。

不同于常规储层,页岩储层以纳米孔为主,具有多种孔隙类型和几何结构,孔隙系统更为复杂,非均质性强^[1]。页岩气以吸附气、游离气及溶解气的形式存在^[5]。了解页岩的孔隙特征,是研究页岩气吸附、解吸和扩散的前提^[1,6]。页岩孔隙的研究方法可分为定性和定量分析2种,前者包括场发射扫描电镜(FE-SEM)、纳米CT等^[7],主要用来观察页岩的孔隙类型和几何形状,受到采样和页岩非均质性的影响,其代表性具有局限性。后者主要是流体注入法,一般包括压汞法和气体吸附法等^[8],虽然该方法可以对整个孔隙系统进行定量评估,但是无法提供孔隙连通性的信息^[9]。前人对页岩孔隙特征的控制因素进行了大量的研究,主要集中在有机碳含量、热演化程度及矿物组成等因素上。如JARVIE等^[2]认为有机孔随生烃量的增加而增多;CURTIS等^[5]的研究表明孔隙随成熟度的提高呈现先增加后减少,最后再增加的复杂变化趋势;MASTALERTZ等^[9]发现黏土矿物含量与微孔体积呈正相关;SHI等^[10]研究发现石英含量的增多有利于大孔的发育,而黏土矿物对中孔发育有一定影响。

以往的研究主要集中在海相页岩^[4],针对海陆过渡页岩孔隙的研究则较少^[11],对其孔隙特征及影响因素的认识仍不够深入。而石炭-二叠系海陆过渡相富有机质页岩在我国广泛发育,其资源潜力巨大^[12]。因此,有必要对海陆相页岩孔隙进行研究。以沁水盆地太原组海陆过渡相页岩为例,通过低温氮气及二氧化碳吸附表征了过渡相页岩的孔隙结构,并进一步探讨了有机碳含量、热演化程度及矿物组成对不同尺度孔隙结构的影响。

1 区域地质背景

沁水盆地位于华北地台中部,山西省东南部,是

一近南北向的大型复式向斜构造盆地^[13-14],盆地四周皆为隆起,其中北邻五台山隆起,南靠中条山隆起,东接太行山隆起,西部为吕梁山隆起和霍山隆起^[15](图1)。晚古生代以前,沁水盆地整体处于构造稳定阶段,之后受印支运动的影响,盆地开始形成。燕山运动使盆地受到强烈的挤压作用,并初步形成向斜构造^[15]。新生代以来,在喜马拉雅运动的影响下,盆地经历了多期构造拉张、挤压作用,最终形成了现今的构造格局^[16]。沁水盆地晚石炭-早二叠世处于海陆过渡环境,以三角洲、潮坪、碳酸盐台地沉积为主^[15,17],发育多套海陆过渡富有机质页岩,由下至上依次为太原组、山西组和下石盒子组^[17]。其中,太原组平均厚度约90 m,岩性包括煤、页岩、砂质泥岩、灰岩、粉砂岩和砂岩。

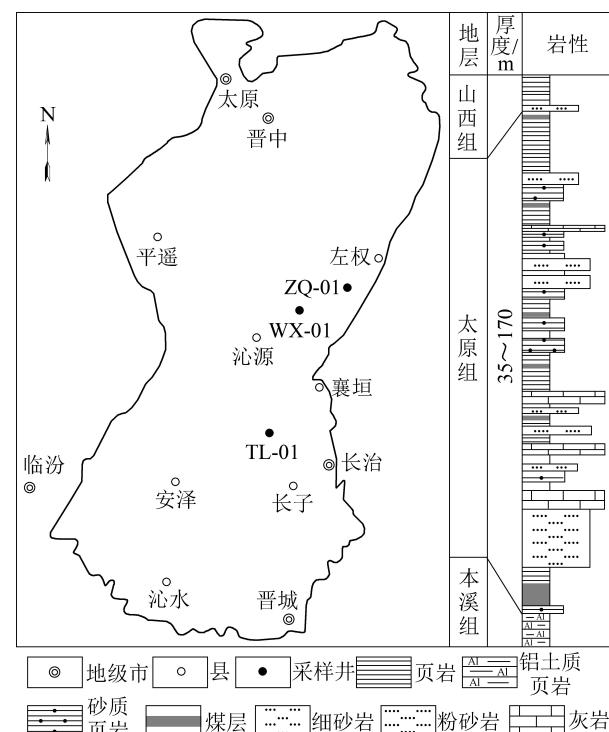


图1 沁水盆地区域位置、井位及太原组岩性柱状
Fig.1 Regional position, well location and Lithologic column chart of Taiyuan Formation in Qinshui Basin

沁水盆地天然气地质调查始于20世纪50年代^[14],自2005年以来在古生界煤层气勘探开发实现了突破性进展,盆地南部现已成为我国目前生产

规模最大的煤层气开发基地^[14]。受煤层的影响,太原组海陆过渡相页岩有机质含量普遍较高,热成熟度较高,页岩气资源丰富^[14~15],是页岩气勘探的重点区域^[12,14]。

2 样品与试验

样品为12个太原组深灰色、灰黑色及黑色页岩,采自沁水盆地三口井,取样位置如图1所示。其中,有机碳测试依据标准GB/T 19145—2003《沉积岩中总有机碳的测定》,测试仪器为LECO CS-230碳硫测定仪。岩石热解分析依据标准GB/T 18602—2012《岩石热解分析》,测试仪器为OG-2000V岩石热解仪。全岩及黏土矿物X-射线衍射分析依据SY/T 5163—2010《沉积岩中黏土矿物和常见非黏土矿物X射线衍射分》,测试仪器为D8 DISCOVER型X射线衍射仪。

场发射扫描电镜(FE-SEM)测试中,首先在抛光仪Leica EMTIC020上借助氩离子束对页岩样品进行抛光处理,将制备好的页岩样品放置于Zeiss Merlin场发射扫描电镜下,对处理后的页岩样品进行二次电子成像。

低温氮气及二氧化碳吸附试验采用的是Autosorb-IQ气体吸附分析仪,在77 K条件下进行了氮气吸附试验,并利用BJH模型计算得到页岩孔隙体积及孔径分布,利用多点BET模型线性回归得到比表面积。在273.15 K下进行了二氧化碳吸附试验,并用DR模型和DFT方程计算分别得到孔隙体积、比表面积及孔径分布。孔径划分采用IUPAC分类方案,即微孔(<2 nm)、中孔(2~50 nm)和大孔(≥50 nm)。

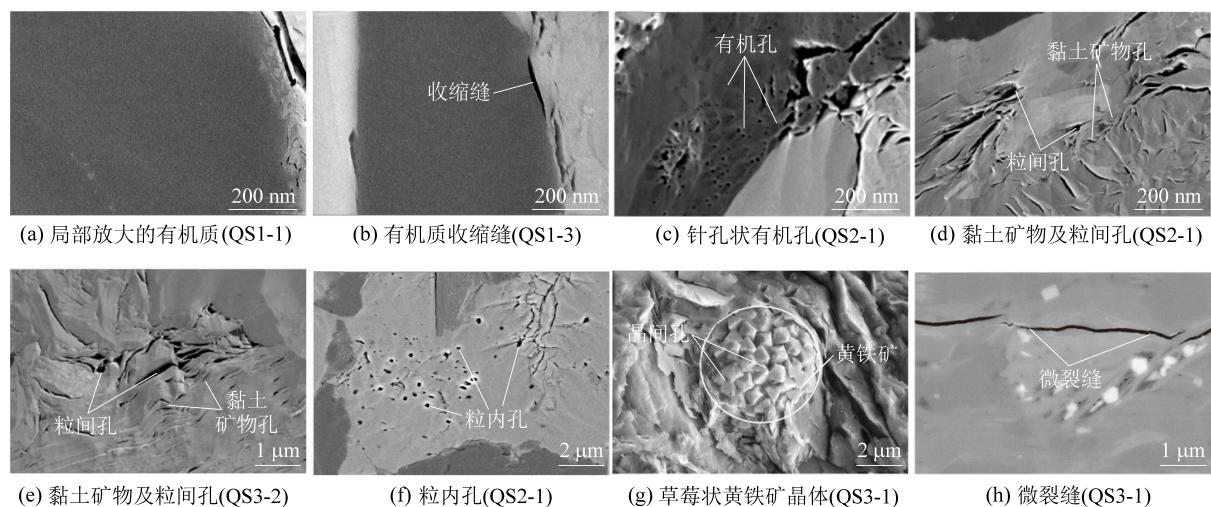


图2 沁水盆地太原组页岩孔隙类型

Fig.2 Pore types in the Taiyuan Formation shales in Qinshui Basin

3 结果与分析

3.1 有机地球化学特征

太原组过渡相页岩TOC质量分数为1.55%~5.21%,平均为3.53%(表1)。所测样品烃潜量(S_1+S_2)为2.09~7.42 mg/g,平均为2.64 mg/g,表明太原组过渡相页岩为优质烃源岩^[18]。岩石热解峰温(T_{max})为406.8~553.5℃,多数为460~500℃,处于成熟到高成熟阶段,有利于页岩气的形成,有机质类型主要为Ⅲ型,以生气为主。

3.2 矿物质组成特征

太原组过渡相页岩主要矿物质成分为黏土矿物和石英,质量分数分别为55%~69%(平均60.83%)和27%~40%(平均33.58%),这与具有高石英含量以及低黏土含量的海相页岩差异较大^[4]。黏土矿物以高岭石和伊利石含量较高,质量分数分别为23%~65%(平均48%)和12%~52%(平均28.67%),其次为伊利石(6%~18%,平均12.58%)和绿泥石(6%~17%,平均10.75%)。

3.3 孔隙类型

页岩中发育不同类型的孔隙,主要分为粒内孔、粒间孔及有机质孔^[6]。扫描电镜观察发现,沁水盆地太原组页岩有机孔不发育(图2a和图2b),仅在少数有机质中可见有机孔,但孔径较小,多为30~50 nm的针孔状孔隙(图2c)。这可能是受到过渡相页岩有机质类型及热演化程度等的制约。太原组页岩普遍发育黏土矿物孔及粒间孔(图2d和图2e),同时存在形成于有机质与黏土矿物等之间的收缩缝(图2d)。黏土矿物中的粒间孔最发育,主要是不规则的层状狭缝和楔形孔隙,一般主要由黏土矿物的取

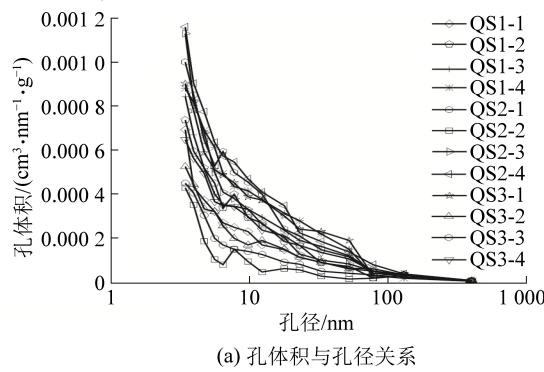
向所限定。如图 5f 所示,太原组页岩中同样发育粒内孔,主要是成岩过程中和成岩后主要由溶蚀作用在脆性矿物颗粒内形成的孔隙,孔隙形态呈多边状、椭圆和近圆状等,孔隙直径为 50~500 nm,而晶间孔则主要发育在草莓状黄铁矿中,孔径变化较大,多在几十到几百纳米之间(图 2g)。另外,在太原组页岩中也可见发育一些微裂缝(图 2h)。

3.3 孔隙结构特征

3.3.1 中-大孔特征

比表面积和孔隙体积是表征页岩储层吸附及储集能力的重要指标。太原组页岩的比表面积和孔隙体积分别为 $1.304 \sim 15.205 \text{ m}^2/\text{g}$ 和 $0.0125 \sim 0.024 \text{ cm}^3/\text{g}$,平均孔径为 $5.73 \sim 8.47 \text{ nm}$,表明页岩主要发育中孔。

图 3 为各样品吸附等温线,该曲线在形态上均呈反“S”型,与 IUPAC 定义的 IV 型等温线相似^[19],且当推动压力 P/P_0 接近 1.0 时,没有表现出吸附限



(a) 孔体积与孔径关系

制,表明太原组过渡相页岩中存在一定数量的大孔或微裂缝。滞回环具有 H3 型滞回环特征^[20],这表明页岩中发育狭缝状孔隙,可能是黏土矿物的片状结构造成的^[19]。由吸附数据求得的孔径和比表面积分布显示,曲线斜率随孔径的增大而减小,且多数页岩的峰值孔径都在 10 nm 以下,表明中孔对孔隙体积和比表面积的贡献大于大孔(图 4)。

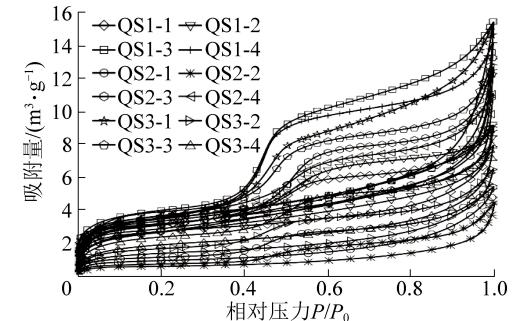
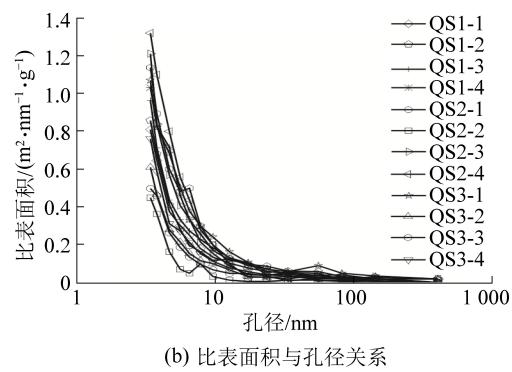


图 3 氮气吸附脱附曲线

Fig.3 Nitrogen adsorption and desorption curves



(b) 比表面积与孔径关系

图 4 中孔和大孔孔隙参数分布

Fig.4 Distribution of pore parameters of mesoporous and macropore

3.3.2 微孔特征

太原组页岩样品二氧化碳吸附曲线类似于 IUPAC 定义的 I 型等温线^[20],均呈略微凸起的形状(图 5)。由二氧化碳吸附测得的样品孔隙体积和比表面积分别为 $0.0021 \sim 0.0051 \text{ cm}^3/\text{g}$ 及 $7.268 \sim 14.762 \text{ m}^2/\text{g}$ (表 1)。孔径分布具有多峰特征,峰值分别出现在 $0.35 \sim 0.40 \text{ nm}$ 、 $0.45 \sim 0.50 \text{ nm}$ 、 $0.50 \sim 0.58 \text{ nm}$ 、 $0.6 \sim 0.70 \text{ nm}$ 和 $0.80 \sim 0.9 \text{ nm}$ 处(图 6a)。比表面积分布与孔隙体积相似,孔径大于 1 nm 的孔对孔隙体积和比表面积的贡献较低(图 6b)。

4 讨论

4.1 过渡相页岩孔隙特征

通过氮气和二氧化碳吸附得到了页岩孔隙体积和比表面积的分布特征^[9]。结果表明,中孔是太原组过渡相页岩孔隙的主要贡献者,占页岩样品总孔隙体积的 $35.6\% \sim 53.7\%$,但其最大比表面积仅为

$4.284 \text{ m}^2/\text{g}$ (表 1),对页岩总比表面积的贡献有限,为 $10.9\% \sim 25.6\%$ 。这表明中孔对游离气丰度有显著的影响,但对吸附气的影响很小。大孔对孔隙体积的贡献仅次于中孔,贡献率为 $25.7\% \sim 40.8\%$,但

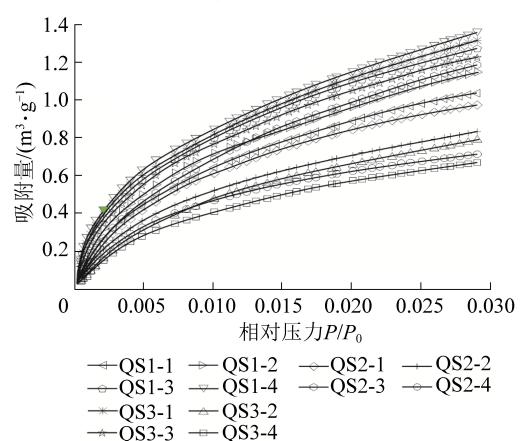
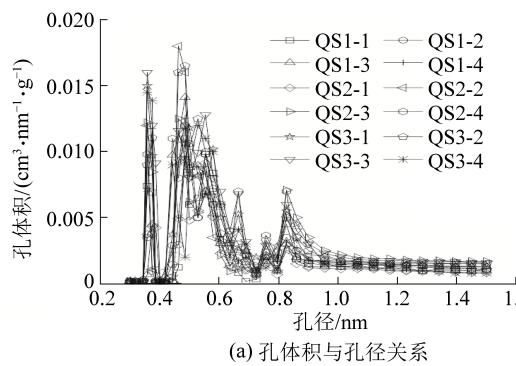


图 5 二氧化碳吸附曲线

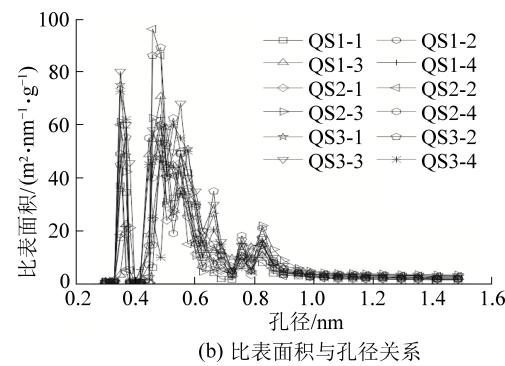
Fig.5 Carbon dioxide adsorption curve

其比表面积仅占总比表面积的1.5%~2.2%,可忽略不计。虽然微孔占总孔隙体积的比例最小(17.1%~25.0%),但其对比表面积的贡献最大,占



(a) 孔体积与孔径关系

总孔隙表面积的72.2%~88.2%,表明页岩中吸附气主要存在于微孔中。



(b) 比表面积与孔径关系

图6 微孔孔隙参数分布
Fig.6 Distribution of pore parameters in micropores

表1 太原组页岩孔体积与比表面积

Table 1 Pore volume and specific surface area of Taiyuan Formation

样品号	孔隙体积/(cm³·g⁻¹)			比表面积/(cm²·g⁻¹)		
	微孔	中孔	大孔	微孔	中孔	大孔
QS1-1	0.003 4	0.008 2	0.004 1	11.021	3.257	0.213
QS1-2	0.003 8	0.006 8	0.005 2	12.167	2.677	0.221
QS1-3	0.005 1	0.010 2	0.005 3	14.762	4.284	0.367
QS1-4	0.004 1	0.010 9	0.008 1	13.564	4.061	0.316
QS2-1	0.003 2	0.006 3	0.005 5	7.268	2.581	0.224
QS2-2	0.003 2	0.005 5	0.005 6	8.341	1.027	0.092
QS2-3	0.004 1	0.006 2	0.007 1	12.412	4.194	0.256
QS2-4	0.002 1	0.006 6	0.003 6	12.512	2.855	0.232
QS3-1	0.004 3	0.009 7	0.005 9	13.962	4.018	0.256
QS3-2	0.003 7	0.006 2	0.004 9	8.576	2.013	0.156
QS3-3	0.003 8	0.007 1	0.006 5	14.741	2.525	0.351
QS3-4	0.002 8	0.006 5	0.003 6	9.453	2.327	0.267

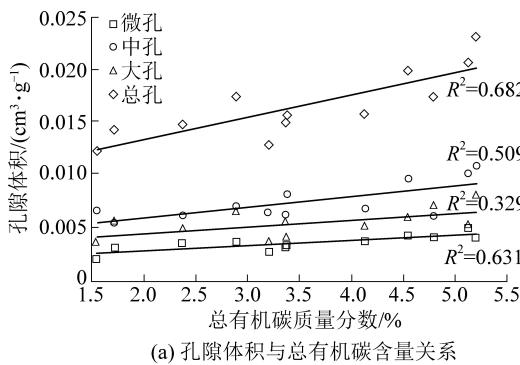
4.2 孔隙发育控制因素

4.2.1 有机碳含量对孔隙结构的影响

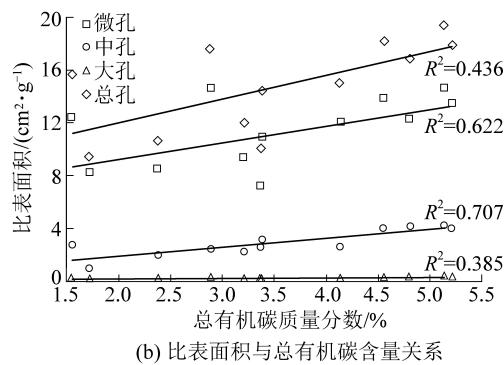
海相页岩孔隙以有机孔为主,且有机碳含量是孔隙发育的主控因素,高TOC含量有利于孔隙发育^[2,21]。而太原组过渡相页岩的TOC含量与孔隙

体积及比表面积的正相关性并不如海相页岩显著,尤其是与大孔隙的正相关性极弱(图7),表明有机质对微孔及中孔有一定影响,但对大孔隙体积的贡献不大。

由于与I型和II型干酪根相比,III型干酪根生



(a) 孔隙体积与总有机碳含量关系

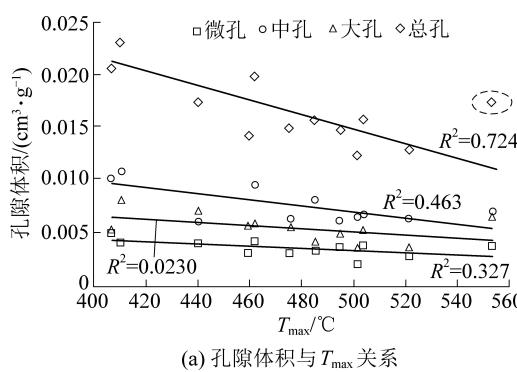


(b) 比表面积与总有机碳含量关系

图7 孔隙体积及比表面积与总有机碳含量关系

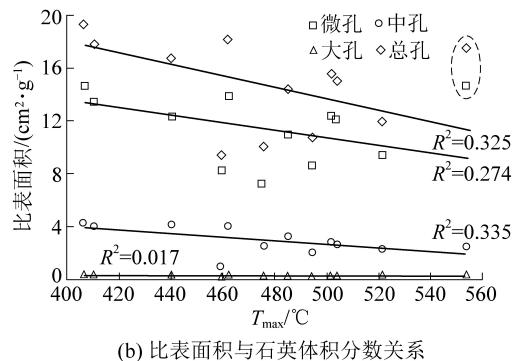
Fig.7 Relationship between pore volume and specific surface area and TOC content

烃速率慢,在成熟过程中挥发分的脱失造成的体积损失量较小^[22]。因此,以往的研究认为过渡相页岩有机孔不发育。有学者研究发现每克有机质对中国南方下志留统和下寒武统页岩孔隙度的贡献率分别为0.72%和0.4%,而对下扬子区二叠纪过渡页岩的贡献仅为0.29%^[21]。INAN等^[23]认为扫描电镜中的可见有机孔仅是其中的一小部分,若仅基于扫描电镜的观察,则页岩的储集能力往往会被低估。然而,由于显微技术分辨率的限制,有机质是否在较小尺度上是多孔的仍然不确定。

(a) 孔隙体积与 T_{\max} 关系

4.2.2 热演化对孔隙结构的影响

有机质的热演化对页岩孔隙体积和比表面积有很大影响。沁水盆地太原组过渡相页岩的各类孔隙体积及比表面积均随 T_{\max} 的增加而减小(图8)。有机质在热演化过程中会生成一定的油气和有机孔,因此,热演化有利于其孔隙的发育。但所测样品多处于高-过成熟阶段^[24],随热演化程度的提高,有机质石墨化加剧,生烃能力降低,且热演化的提高通常意味着埋深及压实作用的增强,这将导致孔隙的闭合或坍塌^[25]。同时热演化对于矿物的生成和转化也具有控制作用^[26],进而控制着了无机孔的增减。



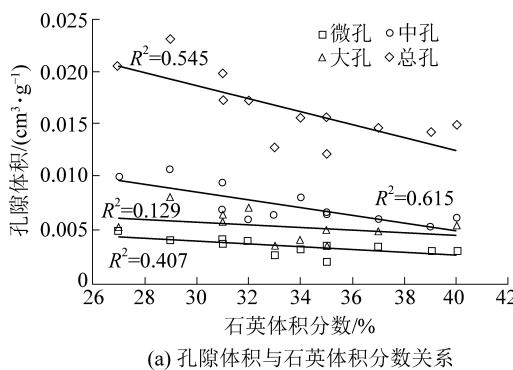
(b) 比表面积与石英体积分数关系

图8 孔隙体积及比表面积与 T_{\max} 关系Fig.8 Relationship between fore Volume and specific surface area and T_{\max}

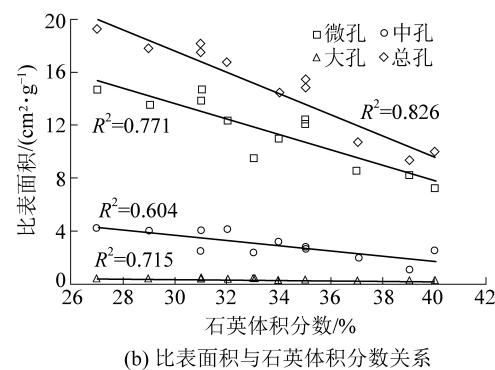
4.2.3 矿物组成对孔隙结构的影响

沁水盆地太原组煤系页岩的石英含量与孔隙体积和比表面积具有一定的负相关性(图9),而海相页岩中石英大多有利于孔隙的发育^[27]。这表明石英含量对孔隙发育的影响与沉积环境密切相关。对于海相页岩,石英等脆性矿物中存在一些原生粒间孔和次生溶孔,对于孔隙体积和比表面积具有一定的贡献^[28]。而沁水盆地太原组页岩中的石英多为

陆源石英,陆源碎屑的大量混入,将大大降低页岩的有机质丰度和黏土矿物含量,因此,石英的富集对孔隙发育具有一定的抑制作用。此外,所研究页岩成岩演化阶段为中成岩至晚成岩期,石英次生加大可达III-IV级,自生石英晶体会向孔隙空间生长堵塞孔隙,导致部分晶间孔隙被破坏。石英含量越高,孔隙堵塞越明显,致使过渡相页岩的孔隙体积和比表面积降低^[29]。



(a) 孔隙体积与石英体积分数关系



(b) 比表面积与石英体积分数关系

图9 孔隙体积及比表面积与石英体积分数关系

Fig.9 Relationship between pore volume and specific surface area and quartz content

黏土矿物可以提供较大的孔体积和比表面积,是孔隙发育的重要控制因素^[8]。太原组过渡相页岩主要由黏土矿物组成,孔体积和比表面积都随着

黏土矿物含量的增加而增加(图10)。黏土矿物对中孔孔体积和比表面积的控制尤为显著,其次是微孔,但对大孔孔体积和比表面积的控制作用较弱

(图10)。

对于黏土矿物的各成分来说,随着高岭石和绿泥石含量的增加,各类孔隙的孔隙体积表现出明显的增加(图11),表明高岭石和绿泥石有利于页岩孔隙的发育,这可能是由于当长石被有机酸溶解产生

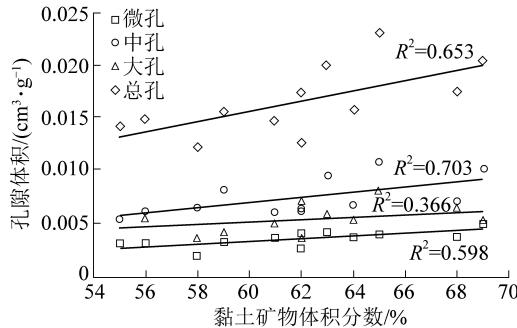


图10 孔隙体积及比表面积与黏土矿物含量关系

Fig.10 Relationship between pore volume and specific surface area and clay mineral content

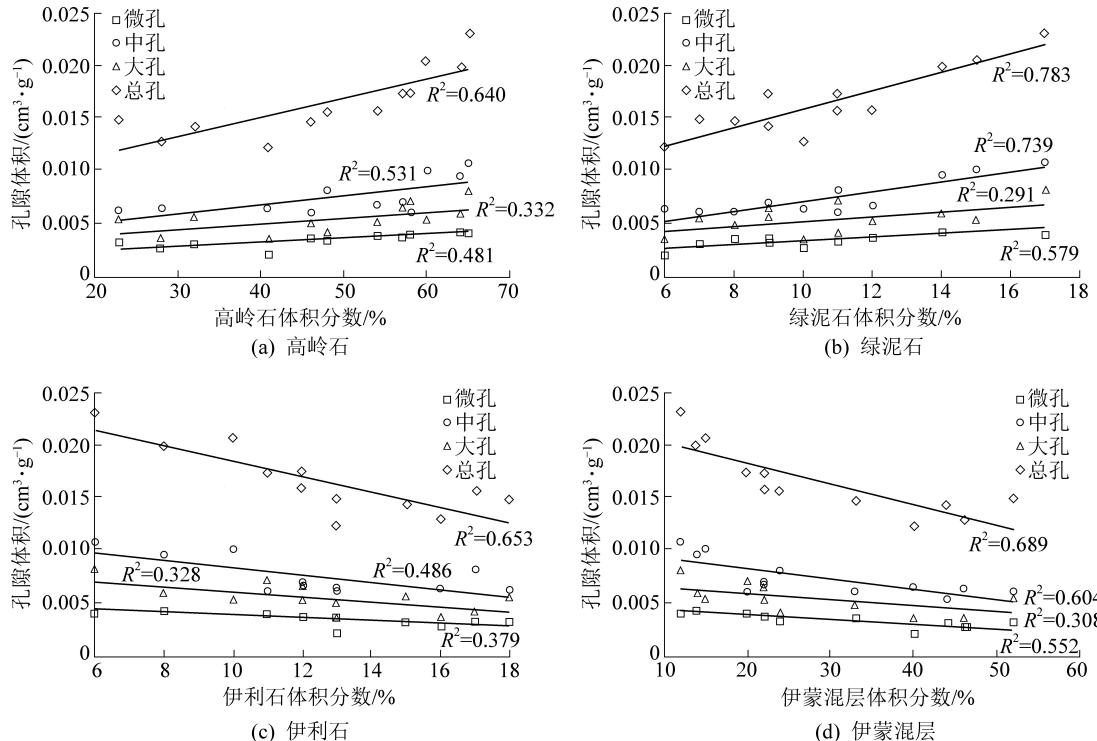


图11 黏土矿物与孔隙体积关系

Fig.11 Relationship between clay minerals and pore volume

5 结 论

1) 太原组煤系页岩样品 TOC 质量分数为 1.55%~5.21%。有机质主要由Ⅲ型干酪根组成,现今多处于高-过成熟阶段。矿物组成以黏土矿物和石英为主,黏土矿物中高岭石和伊蒙混层含量较高,其次为伊利石和绿泥石。

2) 中孔对太原组煤系页岩孔体积的贡献最大(35.6%~53.7%),其次为大孔。虽然微孔对孔隙体

高岭石,孔隙度会随之增加^[30],同时,绿泥石的有序排列也会增加页岩储层的抗压强度和孔隙度^[31]。而伊利石则抑制孔隙网络的发育,这主要是因为蒙脱石在晚成岩阶段转化为伊利石,以及新生成的伊利石会堵塞孔隙从而降低孔隙度^[32]。

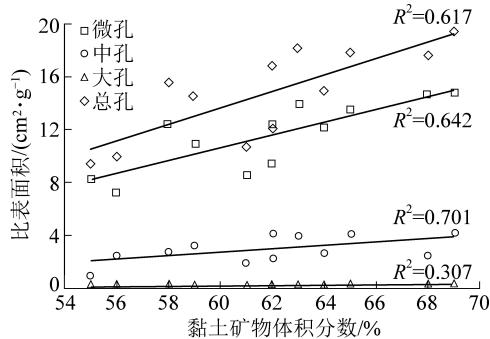


图10 孔隙体积及比表面积与黏土矿物含量关系

Fig.10 Relationship between pore volume and specific surface area and clay mineral content

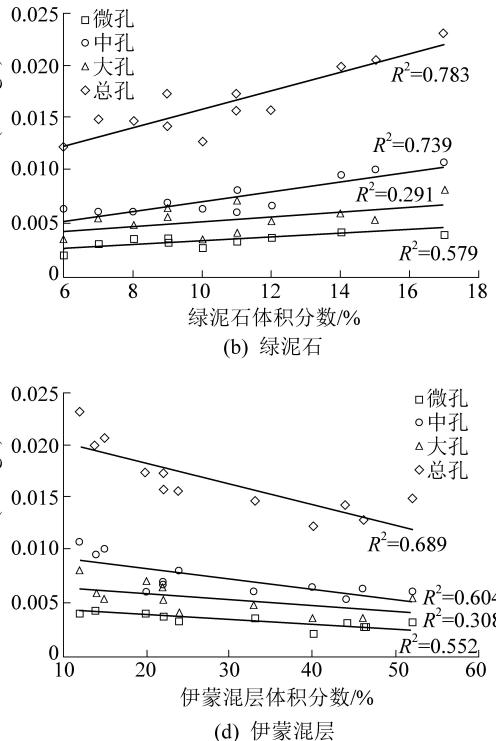


图11 黏土矿物与孔隙体积关系

Fig.11 Relationship between clay minerals and pore volume

积的贡献最小,但其对比表面积的贡献最大(72.2%~88.2%),而大孔对比表面积的贡献几乎可以忽略。

3) 太原组煤系页岩的有机碳含量对微孔及中孔的发育有一定正面影响,但对大孔的影响较小。由于样品多处于过成熟阶段,有机质热演化对孔隙的发育起到了负面作用。

4) 太原组煤系页岩中石英不利于孔隙的发育,而黏土矿物则有利于孔隙的发育,尤其是对中孔发

育的控制作用尤为显著,其次是微孔,对大孔影响较弱。黏土矿物中,高岭石和绿泥石有利于页岩孔隙的发育,而伊利石和伊蒙混层则和孔隙体积呈负相关性。

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