



## The Occurrence and Genesis of Silicified Coals in Tanjung Redeb, Embalut, and Loa Kulu, East Kalimantan, Indonesia

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**Abstract** – The occurrence and genesis of silicified coals in Tanjung Redeb, Embalut, and Loa Kulu have been described and reconstructed by means of petrography and geochemistry. The silicified coals are black, hard, and contain dispersed fine-grained silica minerals. Silica (SiO<sub>2</sub>) contents vary from 58 to 76%, and LOI (mostly represents organic matter) ranges from 23 to 40% of the bulk silicified coal samples based on the ICP-INAA analysis. Microscopic examination shows the presence of quartz, chalcedony, and amorphous opal. Silicification occurs both as layers and nodules within the coal seams. The layer type is usually thin layers (<25 cm) near the top and bottom of the seams, and they can spread horizontally up to 3 m. Nodules occur randomly in the coal seams having a diameter from several centimeters to 1 m. Further microscopic investigation shows that preserved cellular structure is more commonly observed in the layered silicified coal samples. This may suggest that the layered silicification occurred relatively early in the coal seam diagenesis, while the nodule silicification took place during later diagenesis. The coexistence of silica polymorphs in some samples may indicate the presence of multiple silicification stages during coalification.

**Keywords:** silicification, coal, opal, chalcedony, quartz, Kalimantan

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## INTRODUCTION

### Background

Silicification in Indonesian coals have previously been reported, particularly in Tanjung Enim of Muara Enim Formation of South Sumatra Basin (Amijaya and Littke, 2005; Susilawati and Ward, 2006) and in Muara Lawa (Balikpapan Formation, Kutai Basin; Amijaya and Febrianti,

2015). Silicification of coals are also found in some areas in East Kalimantan, such as Tanjung Redeb, Embalut, and Loa Kulu. The silicified coals are locally known as bonecoal, blackstone, or ironstone, which reflect their black colour and hardness. Silicified coals are difficult to recognize which have particular consequences for mining (Lindqvist and Isaac, 1991) because of the damage on excavator buckets and crusher teeth.

Although the presence of silicified coals in Indonesia has been widely reported, the genetic aspects have been less studied. In the present case, the occurrence of silicification in Tanjung Redeb, Embalut, and Loa Kulu coals is reported and discussed. The aim of the study is to describe the occurrence and to reconstruct the genesis of the silicified coals. Geochemical and petrographic analyses were conducted to characterize the chemical and physical characteristics of the silicified coals.

### Geological Settings

The studied areas are located in Tanjung Redeb, Embalut, and Loa Kulu. Geologically, the Tanjung Redeb area is part of the Berau Subbasin within the Tarakan Basin, while the Embalut and Loa Kulu areas are part of the Kutai Basin (Figure 1a). The Tarakan and Kutai Basins are deltaic basins developed since the Eocene in the eastern part of Kalimantan Island (Satyana *et al.*, 1999). The two basins are separated by the Mangkalihat ridge. In the west, the Tarakan and Kutai Basins are bordered by the Kuching High. The northern

boundary of the Tarakan Basin is the Semporna High, while the southern boundary of the Kutai Basin is the Adang Fault.

The Tanjung Redeb coal samples were taken from the Middle Miocene age of Latih Formation (Figure 1b), comprising quartz sandstone, claystone, siltstone, and coal seams with various thickness (0.2 - 5.5 m). The Latih Formation is about 800 m thick and was deposited in deltaic, estuarine, and shallow marine environments (Situmorang and Burhan, 1995).

The Embalut and Loa Kulu coal samples were taken from the Middle Miocene Pulubalang and Balikpapan Formations, respectively (Figure 1b). Pulubalang Fm. is about 900 m thick and was deposited in shallow marine environments, consisting of alternating greywacke and quartz sandstone intercalations with limestone, claystone, coal (up to 4 m) and dacitic tuff (Supriatna *et al.*, 1995). Balikpapan Fm. comprises of alternation of quartz sandstone and clay intercalations with siltstone, shale, limestone, and coal. The formation is 1000 - 1500 m thick deposited in delta front to delta plain environments.

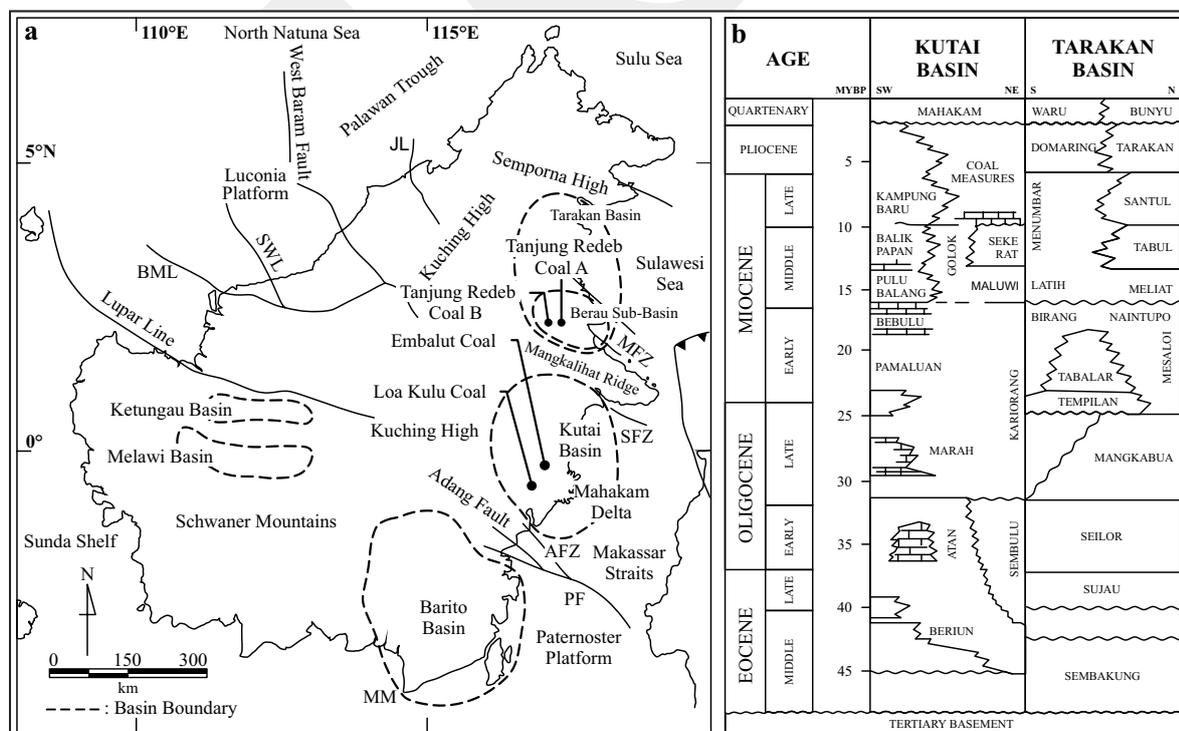


Figure 1. Map of coal basins in Kalimantan Island and locations of the studied areas in Tanjung Redeb, Embalut, and Loa Kulu (modified from Hall *et al.*, 2008) (a), stratigraphic columns of Kutai and Tarakan Basins (Satyana *et al.*, 1999) (b).

## MATERIALS AND METHODS

The samples were taken from freshly exposed rock surfaces of active mine sites in Tanjung Redeb (at two open cast coal mines), Embalut, and Loa Kulu (Figure 1a). Each of the sampling locations is characterized by the presence of multiple coal seams, where some seams are silicified. This study is focused on the silicified coals, coal seams unaffected by silicification and their underlying and overlying sedimentary rocks.

Coal silicification in the studied area typically occurs as either layers or nodules. The first type appears as localized layers commonly near the top and/or bottom of a coal seam (Figure 2a). The thickness of layered silicification in the studied coals range from several centimeters to about 0.25 m and spread horizontally up to 3 m. Silicification of the layer type is present in Tanjung Redeb and Loa Kulu coal seams. The coals are brownish-black, massive, and show rather bright luster with moderate cleat frequency. The silicified intervals exhibit a layered structure and are

relatively duller in luster. The second silicification type (*i.e.* nodules) exhibits a rounded form and can be of various sizes, ranging from several centimeters to about 1 m in diameter (Figure 2b). Nodules can exist in all parts of the coal seams, randomly distributed. Nodule type silicification occurs mostly in Tanjung Redeb and Embalut coal seams. The coals are brownish-black, massive, and have a bright luster with moderate cleat frequency. Higher cleat frequency is usually noted above and below the nodules. The cleats are typically filled by silica mineral assemblages.

Seven silicified coal samples were taken from the three areas, consisting of two samples from each silicification type in the Tanjung Redeb coals, two nodule silicification samples from Embalut coal, and one layered silicification sample from Loa Kulu coal. Additionally, coal seams and the underlying and overlying clastic sedimentary rocks were also sampled. Chip sampling method was applied for the silicified coals, unsilicified coals, and clastic sedimentary rocks. The sampling points are shown in Figure 2c.

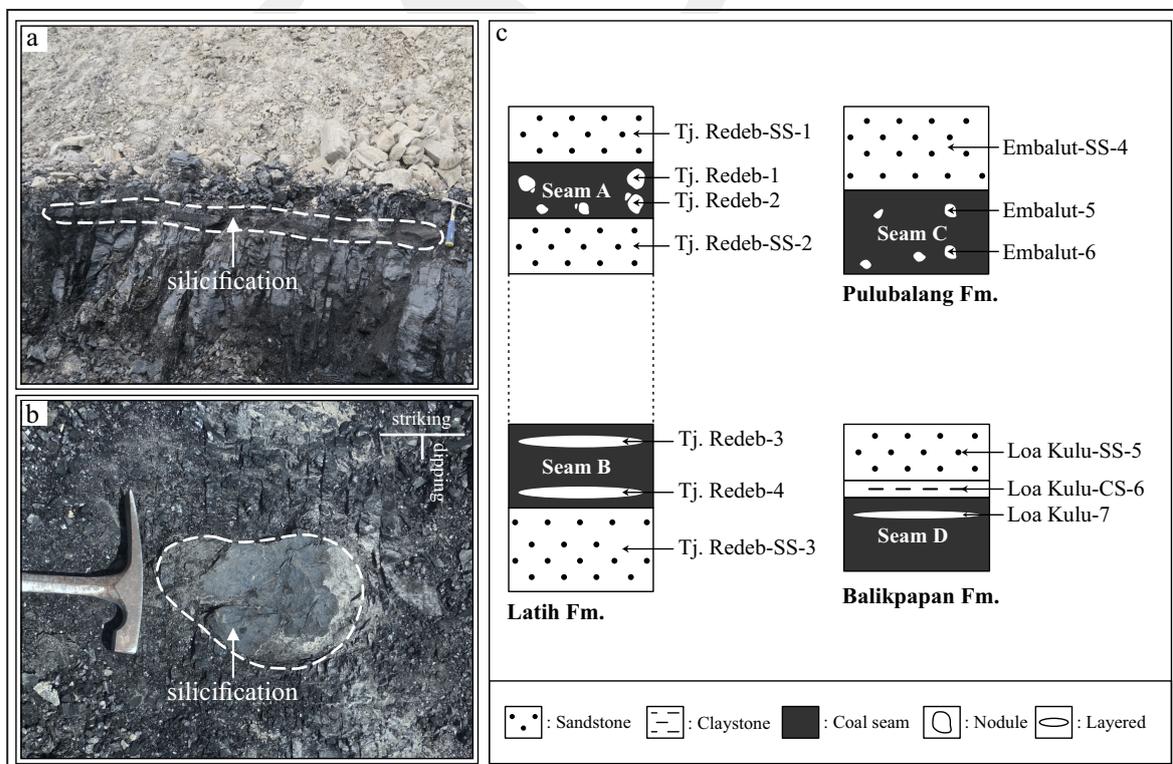


Figure 2. Photographs of layered type silicification in Loa Kulu (a) and nodule type silicification in Tanjung Redeb A. (b), sampling points of unsilicified coal, silicified coal, and the adjacent sedimentary rocks (c).

Geochemical and petrographic analyses were conducted for this study. Prior to analysis, a small amount of sample was ground to obtain -200 mesh particle size. ICP-INAA and X-ray diffraction (XRD) were performed with the pulverized silicified coal and clastic sedimentary rock samples. Proximate and calorific value analyses were applied to the pulverized unsilicified coal samples. ICP-INAA analysis was done in Activation Laboratory (Actlab), Ancaster, Canada, while XRD, calorific value and proximate analyses were carried out in the Research and Development Centre of Mineral and Coal Technology (tekMIRA), Bandung, Indonesia.

Petrographic analysis was performed using a transmitted-light polarization microscope Nikon Eclipse LV100POL to identify transparent to translucent mineral assemblages in thin sections of the silicified coal and clastic sedimentary rock samples. The analysis was also applied using a reflected-light polarization microscope Zeiss Axio Imager A2m to characterize macerals of the coal and silicified coal samples. The sample preparation and maceral analysis follow the

procedures as described by Taylor *et al.* (1998). Sample particles (<1 mm diameter) were embedded in a silicone mold using epoxy resin. The sample pellet was then ground and polished. The microscopic analyses were performed in the Laboratory of Mineralogy, Microscopy and Geochemistry, Institut Teknologi Bandung (ITB), Indonesia. Maceral classification follows ICCP System 1994 (ICCP, 2001; Sýkorová *et al.*, 2005). Scanning electron microscope – energy dispersive spectroscopy (SEM-EDS) was also used in mineral identification to characterize and differentiate silica mineral morphology of the silicified coal samples. The analysis was carried out using SEM instruments JEOL JSM-6510A in the Laboratory of Hydrogeology and Hydrogeochemistry, ITB, Indonesia.

## RESULTS

The XRD analysis shows that quartz is the only crystalline mineral identified in all of the silicified coal samples (Table 1). ICP-INAA

Table 1. Mineralogical and Geochemical Analysis Result

Major Minerals	Tanjung Redeb-				Embalut-		Loa Kulu-	Tanjung Redeb-SS-			Embalut-SS-	Loa Kulu-SS-	
	1	2	3	4	5	6	7	1	2	3	4	5	6
	Quartz	Quartz	Quartz	Quartz, Anorthite	Quartz	Quartz	Quartz	Quartz, Pyrite, Kaolinite	Quartz, Siderite	Quartz, Muscovite	Quartz, Anorthite, Montmorillonite, Cristobalite	Quartz, Calcite, Albite, Cristobalite	Quartz, Kaolinite, Albite, Illite, Montmorillonite
SiO <sub>2</sub>	70.80	74.33	64.93	58.21	76.31	72.65	74.74	59.97	64.27	60.19	68.86	59.16	62.51
Al <sub>2</sub> O <sub>3</sub>	0.04	0.03	0.04	0.02	0.01	0.01	0.02	14.92	13.85	17.89	13.41	13.43	16.85
Fe <sub>2</sub> O <sub>3</sub>	0.09	0.10	0.19	0.29	0.09	0.01	0.12	5.06	4.21	1.91	2.72	3.50	3.82
MnO	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.09	0.07	0.01	0.04	0.14	0.07
MgO	0.04	0.03	0.05	0.05	0.05	0.05	0.05	1.09	0.87	0.85	0.78	1.19	1.52
CaO %	0.08	0.07	0.20	0.27	0.15	0.19	0.20	0.41	0.36	0.18	3.00	7.76	1.82
Na <sub>2</sub> O	0.03	0.02	0.05	0.03	0.03	0.05	0.07	0.57	0.64	0.59	2.94	2.94	2.30
K <sub>2</sub> O	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	1.67	1.66	1.65	1.15	1.32	1.95
TiO <sub>2</sub>	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.724	0.634	0.714	0.425	1.134	0.614
P <sub>2</sub> O <sub>5</sub>	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.08	0.08	0.03	0.12	0.28	0.12
LOI	28.74	25.31	33.62	40.46	22.86	26.87	24.81	14.9	12.09	14.62	5.07	7.73	7.89

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Table 1. Continued....

Major Minerals	Tanjung Redeb-				Embalut-		Loa Kulu-	Tanjung Redeb-SS-			Embalut-SS-	Loa Kulu-SS-		
	1	2	3	4	5	6	7	1	2	3	4	5	6	
	Quartz	Quartz	Quartz	Quartz, Anorthite	Quartz	Quartz	Quartz	Quartz, Pyrite, Kaolinite	Quartz, Siderite	Quartz, Muscovite	Quartz, Anorthite, Montmorillonite, Cristobalite	Quartz, Albite, Cristobalite	Quartz, Kaolinite, Albite, Illite, Montmorillonite	
Au	ppb	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	
Ag	ppm	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	
As	ppm	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	4	6	
Ba	ppm	13	17	59	86	28	28	26	290	235	309	306	342	305
Be	ppm	< 1	< 1	< 1	< 1	< 1	< 1	< 1	2	1	2	1	1	2
Bi	ppm	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2
Br	ppm	< 1	4	2	< 1	< 1	< 1	2	< 1	< 1	< 1	< 1	< 1	< 1
Cd	ppm	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Co	ppm	67	51	19	26	59	26	68	32	51	29	22	28	29
Cr	ppm	< 1	< 1	30	11	5	11	< 1	86	125	147	66	185	85
Cs	ppm	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	10.9	8.6	13	< 0.5	1.1	6.2
Cu	ppm	1	5	< 1	< 1	42	37	53	40	27	36	16	19	27
Hf	ppm	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	2.5	< 0.5	3.3	< 0.5	4.4	4.3
Hg	ppm	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
Ir	ppb	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
Mo	ppm	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2
Ni	ppm	3	4	4	5	9	10	1	55	48	35	26	29	27
Pb	ppm	< 5	< 5	< 5	< 5	< 5	< 5	< 5	17	13	19	7	11	17
Rb	ppm	< 20	< 20	< 20	< 20	< 20	< 20	< 20	100	< 20	140	< 20	< 20	40
S	%	0.185	0.091	0.043	0.051	0.035	0.044	0.052	0.047	0.084	0.035	0.010	0.021	0.130
Sb	ppm	< 0.2	0.4	< 0.2	0.4	< 0.2	< 0.2	< 0.2	1.0	1.0	1.0	0.4	0.3	0.8
Sc	ppm	< 0.1	< 0.1	< 0.1	< 0.1	0.6	< 0.1	< 0.1	15.1	16.4	19.5	6.9	13.4	9.2
Se	ppm	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3
Sr	ppm	10	16	29	39	24	25	24	78	99	79	572	405	290
Ta	ppm	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
Th	ppm	0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	8.5	6.3	11.4	4.3	7.2	8.5
U	ppm	1.0	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	2.0	3.5	2.5	1.3	< 0.5	3.0
V	ppm	< 5	< 5	< 5	< 5	< 5	< 5	< 5	108	92	105	57	143	78
W	ppm	583	427	247	458	325	105	166	115	242	28	48	41	32
Y	ppm	1	< 1	< 1	< 1	3	1	2	24	19	24	9	17	18
Zn	ppm	2	2	2	3	6	8	8	89	83	76	46	116	68
Zr	ppm	2	2	2	< 2	4	4	3	145	142	122	91	200	173
La	ppm	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	25.8	23.1	33.2	17.9	27.4	27.8
Ce	ppm	< 3	< 3	< 3	< 3	< 3	< 3	< 3	49	39	55	35	55	56
Nd	ppm	< 5	< 5	< 5	< 5	< 5	< 5	< 5	30	13	23	6	44	12
Sm	ppm	< 0.1	< 0.1	< 0.1	< 0.1	0.1	< 0.1	< 0.1	3.9	3.0	4.1	2.5	4.0	3.8
Eu	ppm	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	0.9	< 0.1	0.8	0.8	1.1	0.9
Tb	ppm	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Yb	ppm	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	2.0	1.8	2.2	0.8	2.3	1.9
Lu	ppm	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	0.28	0.35	0.48	0.15	0.31	0.29

analysis indicates SiO<sub>2</sub> contents are very high (58 – 76%), and the other oxides are very low (<0.5%), suggesting that silica minerals dominate the samples. The high content of LOI (23 –40%) may relate to the organic matter within the silicified coal samples. The concentration of MnO, K<sub>2</sub>O, TiO<sub>2</sub>, P<sub>2</sub>O<sub>5</sub>, and most trace elements in the silicified coal samples are below detection limit. In the sandstone and claystone samples, quartz is the most dominant crystalline mineral as indicated by the XRD results. Other minerals found in the samples include feldspar (albite, anorthite), clay minerals (kaolinite, montmorillonite, illite), pyrite, and carbonate minerals (siderite, calcite). The ICP-INAA analysis confirms the presence of significant aluminosilicate minerals in the clastic sedimentary rocks as indicated by the higher content of Al<sub>2</sub>O<sub>3</sub> (typically >13%; Table 1).

Transmitted-light polarization microscopy reveals that quartz is not the only mineral found in the silicified coal samples. Chalcedony is commonly found in the Tanjung Redeb samples as indicated by its fibrous structure. This mineral might be identified as quartz by the XRD analysis, as both minerals show similar diffraction patterns (Mustoe, 2015). Opal exhibits complete extinction by crossed-polarized light microscopy. Table 2 summarizes the silicification occurrence and its microscopic characteristics in the samples.

Under transmitted-light microscopy, a relic cellular structure is usually found in layered type silicifications as appears in Tanjung Redeb-3, Tanjung Redeb-4 (Figure 3a), and Loa Kulu-7 (Figures 3b-c). Quartz and chalcedony commonly fill voids and replace cell walls, respectively. Opal showing lepisphere structure mostly fills cell voids. Figure 3a demonstrates obvious cell wall replacement by chalcedony and cell lumen filling by lepisphere opal. Broken relic cellular structure is commonly shown by nodule silicification in samples of Tanjung Redeb-1 (Figures 3d-e), Tanjung Redeb-2 (Figure 3f), Embalut-5 (Figure 3g), and Embalut-6 (Figure 3h). Opal aggregates fill voids in sample Tanjung Redeb-2 (Figure 3f). Most of cracks and voids in the Embalut samples are filled by quartz (Figure 3g). In the Embalut-6 sample, quartz aggregates are frequently present in association with thin organic matter as indicated by Figure 3h. Further investigation using fluorescence light suggests that the organic matter is liptinite maceral. It shows unspecific forms that are usually associated with coarse quartz aggregates.

The reflected-light microscopic analysis reveals that the Tanjung Redeb, Loa Kulu, and Embalut coals are dominated by huminite macerals (>80%, volume), and contain lower amounts of inertinite and liptinite macerals (Table 3, Figures

Table 2. Occurrence of Silicification and Its Microscopical Characteristics in Tanjung Redeb, Embalut, and Loa Kulu Coals

Sample	Occurrence – silicification type	Microscopical Characteristics
Tanjung Redeb-1	In middle of coal seam - nodule type	Principal minerals: opal, chalcedony, quartz. Relic cellular structure is rarely observed, opal fills voids, chalcedony are dispersed in organic masses, quartz usually fills open spaces or cracks.
Tanjung Redeb-2		
Tanjung Redeb-3	Near roof of coal seam - layered type	Principal minerals: opal, chalcedony, quartz. Relic cellular structure is frequently appeared. Chalcedony commonly replaced cell walls. Opal typically fills cell voids, while quartz generally fills voids and cracks.
Tanjung Redeb-4		
Embalut-5	In middle of coal seam - nodule type	Principal minerals: quartz. Relic cellular structure is rarely present. Quartz crystals are dispersed in the organic masses and fill cracks and voids. Coarse quartz crystals in association with liptinite are often observed.
Embalut-6		
Loa Kulu-7	Near roof of coal seam - layered type	Principal minerals: quartz. Relic cellular structure is commonly observed. The quartz crystals fill voids including cell lumens or replace cell walls.

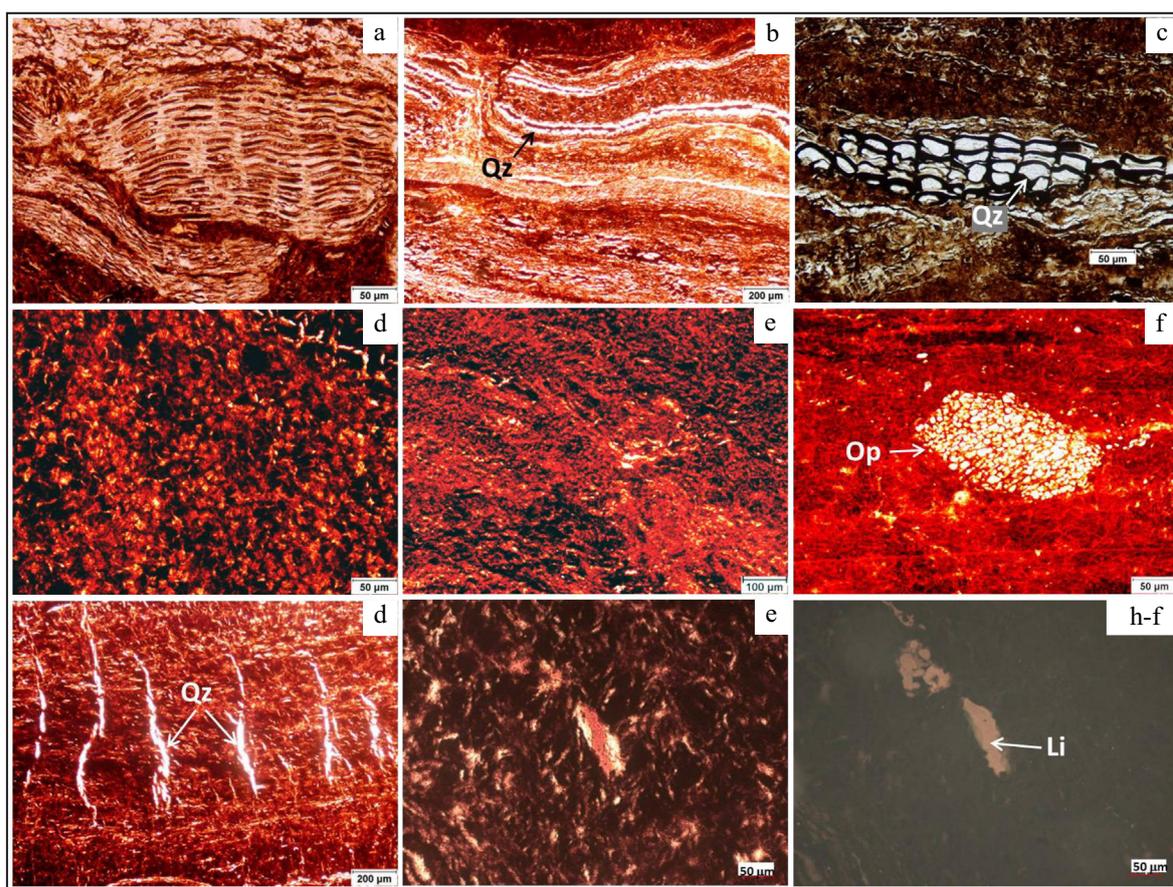


Figure 3. Photomicrographs of silicified coal samples. Relic cellular structure is obviously seen in Tanjung Redeb-4 (a) and Loa Kulu-7 (b,c) samples. Cell wall replacement by chalcedony and cell lumen filling by opal are observed in Tanjung Redeb-4. Quartz commonly fills voids including cell lumens in Loa-Kulu-7. Destroyed relic cellular structure is mostly found in Tanjung Redeb-1 (d,e), Tanjung Redeb-2 (f), Embalut-5 (g), and Embalut-6 (h). Quartz is dispersed in organic masses or fills cracks. Opal aggregate is seen in Tanjung Redeb-2. Coarse quartz crystal frequently appears in association with liptinite maceral in Embalut-6. Note: F (fluorescence light microscopy): Qz (quartz), Op (opal), Li (liptinite).

Table 3. Results of Chemical and Petrographic Analyses of Coal Samples

Parameter	Coal Sample			
	Seam A	Seam B	Seam C	Seam D
Huminite (% v)	83.6	88.6	81.4	80.8
Inertinite (% v)	4.6	3.4	4.8	6.0
Liptinite (% v)	4.8	2.8	6.2	9.2
Mineral (% v)	7.0	5.2	7.6	4.0
Ro (%)	0.47	0.45	0.45	0.46
Calorific value (cal/g, adb)	5,774	5,449	5,458	5,405
Inherent moisture (% adb)	12.50	14.18	16.86	16.83
Ash (% adb)	4.87	3.62	6.51	3.75
Volatile matter (% adb)	38.93	39.92	34.79	38.14
Fixed carbon (% adb)	43.70	42.28	41.84	41.28

4a-d). Huminite reflectance (Ro) of the coals are about 0.45%. The calorific values of the coals range from 5,400 to 5,800 cal/g (adb). The proximate analyses indicate that the coals generally

contain relatively moderate amounts of inherent moisture (about 12.5 - 17%, adb) and volatile matter (about 35 - 40%, adb). These data indicate that the coals are subbituminous.

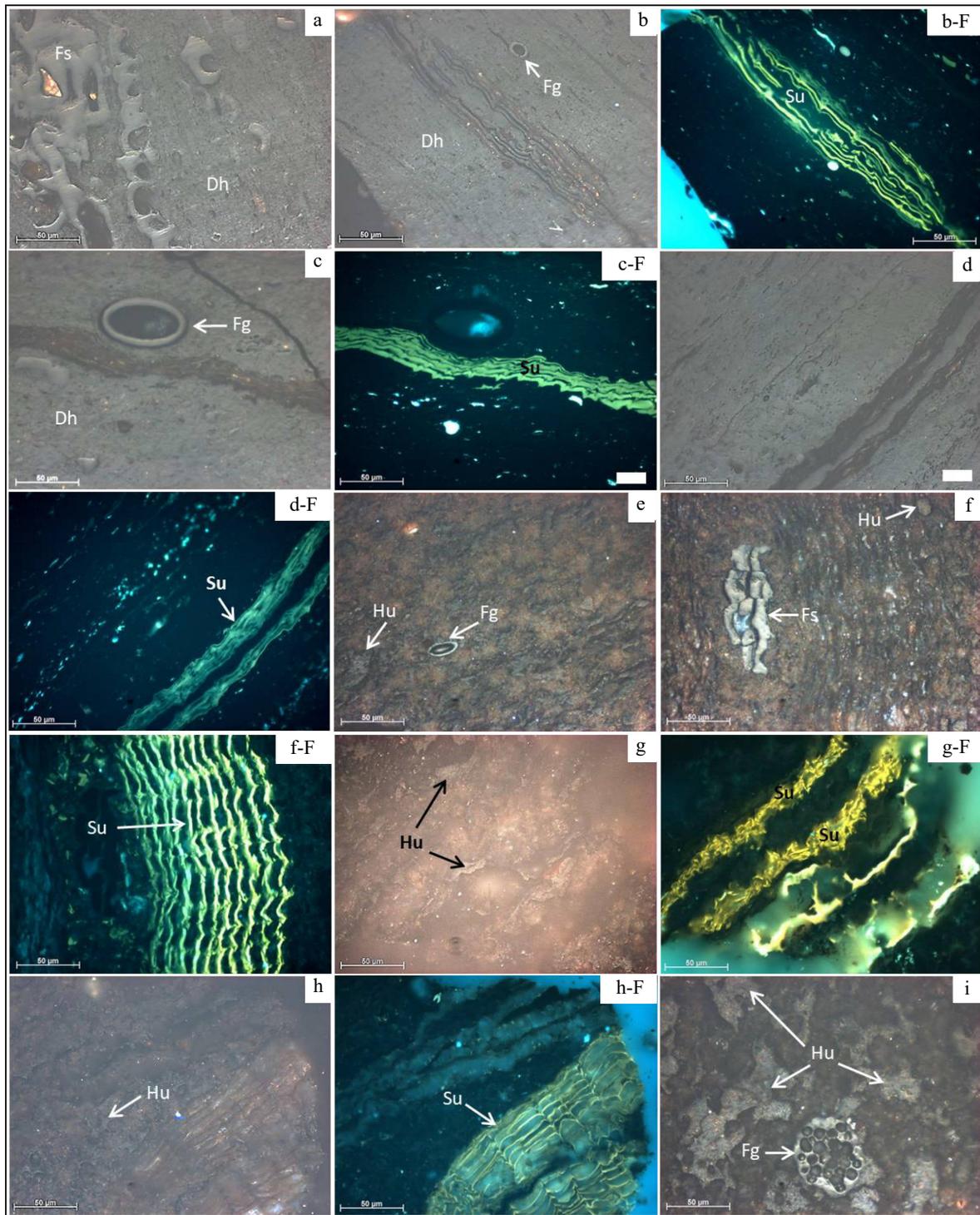


Figure 4. Photomicrographs of the coal seam (Tanjung Redeb A, Tanjung Redeb B, Embalut C, Loa Kulu D) samples (a-d, respectively) and the silicified coal (Tanjung Redeb-1, Tanjung Redeb-4, Embalut-5, Loa Kulu-7) samples (e, f, g, h, respectively). Silicification tends to occur in huminite rather than in inertinite and liptinite macerals. Note: F (fluorescence light microscopy), Dh (detrohuminites), Hu (huminites), Fs (fusinites), Fg (funginities), Su (suberinites), scale bar represents 50 µm length.

The examination of polished sections of silicified coal indicates that most huminite macerals could not be obviously observed. The maceral

body has been replaced by silica minerals (Figures 4e-i). Although inertinite and liptinite macerals account for only minor amounts in the silicified

coals, most of their whole bodies can still be identified under the microscope. Fusinite (Figure 4f) and especially funginite (Figures 4e,i) are common intact inertinite macerals observed within the samples. Liptinite macerals found in the silicified coals consists of suberinite (Figures 4f-h), sporinite, and liptodetrinite. It appears that the silicification has a preference to occur within huminite rather than inertinite and liptinite macerals.

Further examination of the silicified coal samples using SEM-EDS analysis confirms the presence of various silica minerals. Chalcedony shows distinct fibrous internal structure and sometimes exhibits botryoid surface structure (Figures 5a-c). Quartz is recognized from its hexagonal form (Figures 5d-f). Opal is identified by its lepisphere structure (Figures 5b-c). In petrified

woods, opal may be opal-A (amorphous) or opal-CT (incipient crystallization of cristobalite and tridymite) (Mustoe, 2015). In the silicified coal samples, distinguishing opal-A and opal-CT using polarized light microscopy was unreliable. SEM analysis reveals all opal is smooth and shows no microcrystalline texture. This may suggest that the opal is amorphous.

The petrographic investigation of the Embalut and Loa Kulu sandstones found volcanic rock fragments with fine grained porphyritic texture as shown in Figure 6a. The fragments typically exhibit a weathering texture in the boundaries due to replacement of the microcrystalline silica and glass by calcite. In addition, feldspars in the sandstones also exhibit weathering (Figure 6b). The Tanjung Redeb sandstones contain fragments

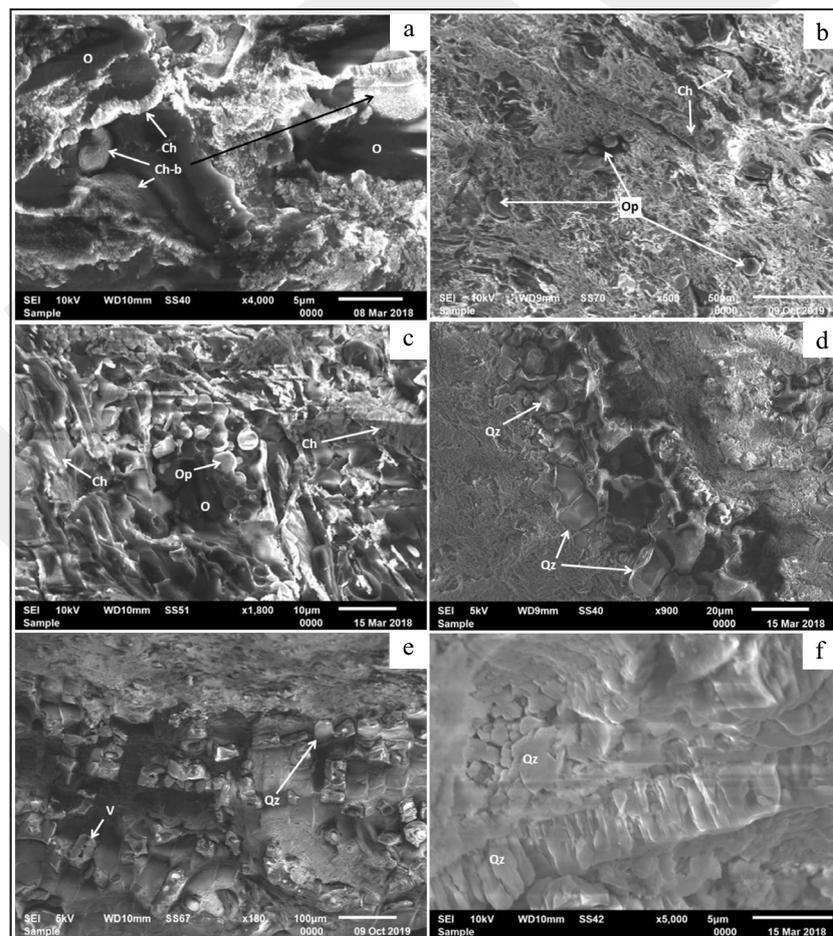


Figure 5. SEM photomicrographs of the silicified coal samples Tanjung Redeb-4 (a-b), Tanjung Redeb-1 (c-d), Loa Kulu-7 (e) and Embalut-5 (f). Note: O (organic matter), Ch (chalcedony), Ch-b (chalcedony showing botryoidal surface structure), Op (opal), Qz (quartz), V (void).

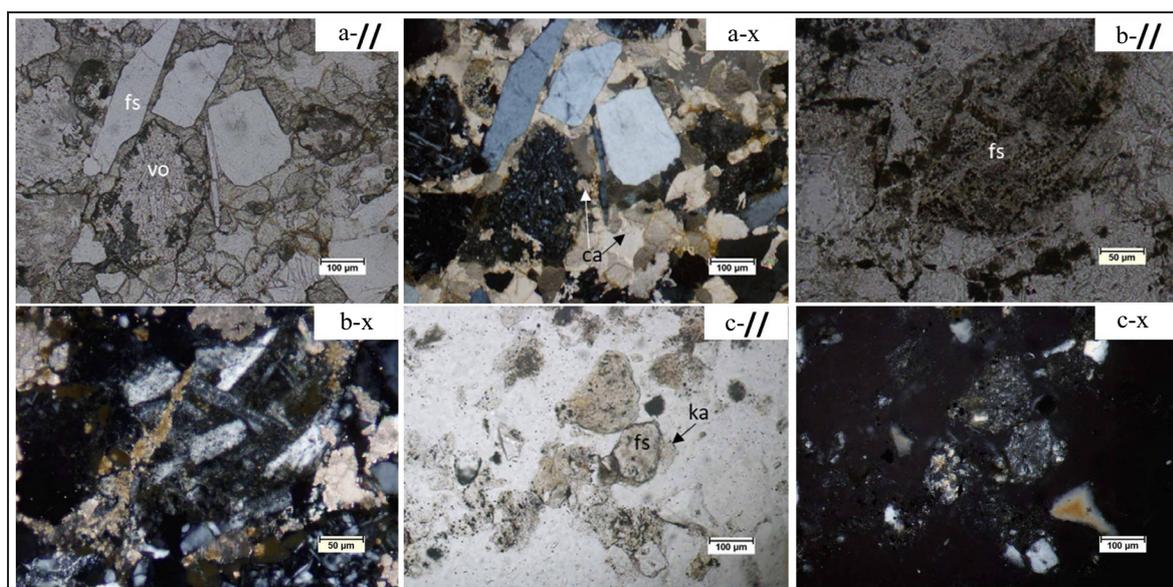


Figure 6. Volcanic rock fragments (vo) found in Embalut sandstone (Embalut-SS-4) mostly exhibit alteration and replaced by calcite (ca) along its boundary (a). Alteration of feldspar minerals (fs) of igneous rock fragments observed in Loa Kulu sandstone (Loa Kulu-SS-5, b). Severely altered minerals, including decomposed feldspars to kaolinite (ka), and loose fragments are commonly found in Tanjung Redeb sandstone (Tanjung Redeb-SS-2, c). Note: // and x indicate parallel and crossed nicol configurations, respectively.

that are typically loose and severely altered (Figure 6c). Most minerals, including feldspar, may have already been altered to kaolinite.

## DISCUSSION

### Silica Source of The Silicified Coals

Silica in coal can be present in three forms: detrital, early-, and late-diagenetic. Detrital silica comes from sediment brought in by water and/or air transportation. Detrital silica, due to transportation, is usually rounded. Early diagenetic silica in coal is precipitated from pore fluids during early burial when cleats have not been developed. Late diagenetic silica is precipitated in cleats and fractures or replaces coal and earlier mineral phases after cleats were developed (Lindqvist and Isaac, 1991; Sykes and Lindqvist, 1993). In the present case, detrital silica is absent in the silicified coals as no rounded silica clasts were observed in the samples. Under the microscope, the silica minerals mostly exhibit massive texture and are interlocked with organic matter (Figure 3), suggesting diagenetic silicification.

Silica in diagenetically silicified sedimentary rocks may be sourced from a few origins. Sykes and Lindqvist (1993) and Ward (2016) suggested that silica could derive from chemical weathering of silicate minerals in surrounding basement rocks carried into the buried peat/coal by groundwater during early diagenesis. Alteration of feldspars from sandstones could also result in dissolved silica which is further transported within the sandstones or into the underlying and overlying rocks including coal during late diagenesis (Sykes and Lindqvist, 1993; Rezaee and Tingate, 1997). Silica may also originate from transformation of clay minerals. During burial, smectite is altered to illite, resulting in significant amounts of silica in solution (Weaver, 1959; Środoń, 1999; Peltonen *et al.*, 2009). Dai *et al.* (2008, 2014) reported the presence of abundant authigenic quartz in Xuan Wei coals. This mineral was attributed to siliceous hydrothermal solution origin, which precipitated during peat accumulation or at least before significant compaction. Additionally, Wüst *et al.* (2008) demonstrated that Al-Si-rich phytoliths of forest peat were transformed into new minerals mainly euhedral quartz and clay

minerals during artificial coalification. Statistical and microscopic analysis have also suggested that authigenic quartz may originate from biogenic grains such as phytoliths and chrysophytes cysts (Cecil *et al.*, 1981; Ruppert *et al.*, 1985; Ruppert *et al.*, 1993). This indicates that minerals could derive from inorganic elements that were present in the plant biomass without any addition from terrigenous sources during coalification.

In the current study, silica source from smectite illitization is less likely. The coal seams are underlain and overlain mostly by sandstones, which have a limited amount of clay minerals. Overlying thin claystone is observed in the Loa Kulu coal seam. However, illite in this claystone accounts for only a minor amount. Smectite illitization commonly occurs during catagenesis or metagenesis phases where the burial temperature is above 100°C (Hoffman and Hower, 1979; Polastro, 1993). Considering the relatively low rank of the investigated coals (subbituminous), smectite illitization of the sandstones and claystone is not thought effectively occurred yet.

Authigenic quartz originating from hydrothermal solution observed in Xuan Wei coals reported by Dai *et al.* (2008, 2014) occurs together with chamosite, which is a product of hydrothermal alteration. In the present study, chamosite or other hydrothermal-related minerals are not detected. The coals were deposited in deltaic basins where hydrothermal activities are not observed in the investigated area and its surroundings. Therefore, silica derivation from hydrothermal fluids in the coals might not be the case.

Phytoliths may be insignificant for the occurrence of silicified coals. Wüst and Bustin (2003) suggested that plants could uptake some elements, primarily Al, Si, and Fe, in tropical wetland environments through their root system, leading to phytolith formation within the plants. In such case, further coalification processes should therefore transform the phytoliths into both quartz and clay minerals. However, the present study reveals that minerals in the silicified coals will almost exclusively consist of silica minerals. Additionally, if there is transformation of silica

from phytoliths, it would be expected to have a homogenous distribution within the coal seam. In contrast, the silica is concentrated as nodules or occur as distinct layers. Thus, it is suggested that the silica of the silicified coals have not originated in any significant way from phytoliths.

Silica source from weathering of surrounding basement rocks may be unlikely. In most Indonesian basins, coals occur as multiple seams with variable thickness. In the areas studied for this paper, only some coal seams are silicified. If the silica derives from basement weathering, then silicification is expected to occur throughout all coal seams, rather than limited to a few. This therefore leads to an interpretation that the silica might be significantly related to the underlying and/or overlying sedimentary rocks.

Further investigation supports that silica responsible for the coal silicification might originate mostly from the underlying and overlying sandstones. Common replacement of volcanic rock fragment boundaries by calcite in the Embalut and Loa Kulu sandstones is evident (Figure 6a). Wakizaka (1998, 2000) demonstrated that reactive minerals (tridymite, cristobalite, and glass) of volcanic rocks would readily dissolve in alkaline condition. He also suggested that those sedimentary and metamorphic rocks could dissolve, primarily due to the presence of low crystallinity minerals (very fine silica). In the current study, alkaline conditions that may have accelerated the dissolution of microcrystalline silica and glass of the volcanic rock fragments is implied by a relatively higher Ca content in the sandstones (obtained from ICP-INAA analysis, Table 1) in the form of calcite cement observed by petrographic examination.

All the studied sandstone samples indicate alteration of feldspar especially in the Tanjung Redeb coal seams. Alteration or dissolution of feldspar has been previously discussed to occur in sandstones, which is also responsible for forming secondary porosity during diagenesis (Siebert *et al.*, 1984; Bevan and Savage, 1989; Taylor *et al.*, 2010). Gudbrandsson *et al.* (2014) demonstrated that plagioclase could dissolve in any conditions

where typically the dissolution rates increase with decreasing pH in acid conditions, or with increasing pH in alkaline conditions. During plagioclase dissolution, silica would be released into solution.

Ruppert *et al.* (1993), Li *et al.* (2001), and the references therein discussed that minerals can be altered during peatification and coalification stages. In peatification stages, minerals are decomposed by the presence of organic acids and/or *in situ* microorganisms (Andrejko *et al.*, 1982; Bustin and Lowe, 1987). Furthermore, Hiebert and Bennett (1992) demonstrated that indigenous bacteria altered silica and silicate minerals significantly in a shallow petroleum-contaminated aquifer. During early diagenesis, minerals continued to be decomposed because of organic acid complexations and higher temperature. In the late coalification stage, mineral alteration is further advanced primarily by higher formation temperature.

In the present case, organic acids, and probably certain microorganism if still exist, might also have a role in mineral dissolution. Such a mechanism could take place in the transition rocks between the coal seam and the underlying or overlying clastic rocks, which are often carbonaceous. Organic matter in the transition rocks might produce organic acids during diagenesis and contribute in mineral dissolution.

The silica solution produced from alteration of microcrystalline silica of volcanic rock fragments and dissolution of feldspar might further be transported into the coal seam, promoting silicification within the layer. The more intensive the mineral alteration and dissolution in the underlying and overlying rocks, the more possibility of silicification in the coal seams. Intensive alteration of the Tanjung Redeb sandstone is clearly observed, both megascopically and microscopically. The sandstone is weak and could be easily broken using a hammer. Under the microscope, the sandstone fragments including feldspar are intensively altered, primarily to kaolinite (Figure 6c). This observation agrees with the intensity of silicification of the Tanjung Redeb coal seams, especially Seam A, which is much higher when compared with those of the Embalut and Loa Kulu areas.

### Genetic Model of Silicified Coals

Permineralization and replacement are the main processes in wood petrification (Mustoe, 2017). Permineralization develops when original cellular material becomes entombed as voids, including cell lumens, filled with silica or other minerals. Replacement takes place when original organic constituents such as cell walls degrade during fossilization. Mustoe (2017) suggested that permineralization and replacement may not mutually exclusive, as the processes could happen simultaneously during diagenesis. Both permineralization and replacement in wood would commonly preserve cellular structures. However, when the organic matter has been destroyed during coalification, cellular structures could not be represented by further mineralization. The degree of cellular structure preservation of the mineralized organic matter, therefore, could be used to imply the relative timing of mineralization during diagenesis (Sweeney *et al.*, 2009). In the present case, layered silicification indicates better preservation of cell structure, whereas poor cell structure preservation is indicated by nodule silicification. This suggests that layered silicification might have occurred relatively early in the diagenesis process, while nodule silicification might happen in the later stages of diagenesis.

Silicification in woods and coals is common, because silica has an affinity with plant organic matter especially lignin and hemicellulose (Leo and Barghoorn, 1976; Mustoe, 2015). During peatification, lignin and hemicellulose would transform to huminite in a reducing environment. When an environment is relatively oxic or moist, oxidation may occur and lignin and hemicellulose would transform into inertinite macerals (Taylor *et al.*, 1998). In the studied samples, silicification tends to occur in huminite macerals rather than in inertinite and liptinite macerals. Although inertinite macerals originate from lignin and hemicellulose, these macerals are less reactive due to fusinitization prior to deposition or during peatification (Taylor *et al.*, 1998). Liptinite macerals have different precursors. They originate as resins and waxes of plants. The inertinite and

liptinite macerals may therefore be more resistive to silicification.

Wood and coal silicifications commonly involve polymorphs of opal, chalcedony, and quartz. The mineralization may be initiated with primary precipitation of opal-A from silica solution, which further transforms to opal-CT, chalcedony, and eventually quartz as diagenesis progresses (Williams and Crerar, 1985; Williams *et al.*, 1985; Mustoe, 2017). However, coexistence of the polymorphs in silicified sedimentary rocks does not always indicate a diagenetic pathway. Mustoe (2015) and Viney *et al.* (2016) discussed that all the polymorphic minerals could be primarily precipitated during wood petrification. Thus, multiple silicification pathways could take place during the silicification of wood and coal.

Microscopic investigations reveal common coexistence of silica polymorphs in the studied Tanjung Redeb samples (Table 1; Figures 3a, 5b-c). Tanjung Redeb-4 sample (Figures 3a, 5a-b) shows the occurrence of chalcedony where its texture represents cell walls. The cell lumens are infilled by loose opal grains. It can be interpreted that there are at least two silicification stages. First, silica solution might permeate into the coal and precipitate silica through cell walls replacement. Second, later permeation of silica solution might precipitate opal in the empty cell lumens. The former silicification may represent replacement during relatively early coal diagenesis, resulting preserved relic cellular structure. The existence of chalcedony showing fibrous internal structure and botryoid surface structure (Figure 5a) suggests a diagenetic transformation of opal to chalcedony.

Permineralization is likely present in the Loa Kulu coal as revealed in Figure 3c. Preserved relic cellular structure is observed where the cell lumens are infilled by quartz and the cell walls remain unreplaced. Figure 5e confirms the silica permineralization in the Loa Kulu coal, although quartz is not in a hexagonal form. This may be due the mineral would rather reflect cell lumen morphology as its external form. The presence of voids in quartz (Figure 5e) implies that the

mineral developed through silica precipitation which began on the surfaces of cell walls. This initial silicification would form a template for reflecting cell lumen morphology. Such organic templating mechanism has been previously well documented in wood permineralization by Leo and Barghoorn (1976) and Mustoe (2017). Further silica growth would be inward, towards the centre of each cell lumen. In the current case, the silica might be either opal/chalcedony that further transformed during diagenesis to quartz or primary precipitated quartz.

Figure 7a (left) and b (right) illustrate reconstructed models representing silicified coal genesis in the studied areas for both layered and nodule silicification types, respectively. Initially, both models developed peats (Figures 7a1 and 7b1). Further diagenesis resulted in coal seams where organic matter had not been physically severely degraded, so the cellular tissues were still mostly preserved (Figure 7a2). In the left model, the underlying and overlying clastic rocks underwent alteration, resulting silica solution that migrated to the coal seam. The seam may not yet have been compacted fully and contained significant porosity, allowing permeation of the solution into the coal. Permineralization or replacement processes then occurred once the solution reached the top or bottom of coal seam. Due to larger reactive surface area of the porous coal, the mineralization sites were likely limited by proximity to the silica solution sources. Mineralization in early diagenesis would result in layered of macro structures and in preserved cellular microstructure. Multiple silicification phases probably occurred, which resulted in the coexistence of chalcedony replacing cell walls and opal infilling cell lumens. In Figure 7b2, the adjacent rocks might have not been altered yet, so there was no silica solution promoting coal silicification.

Further coalification generated more compacted coals and cleats (Figures 7a3 and 7b3). In Figure 7a3, the alteration in adjacent rocks might be less intense, so further silicification may not have been significant. Subsequently, the coal

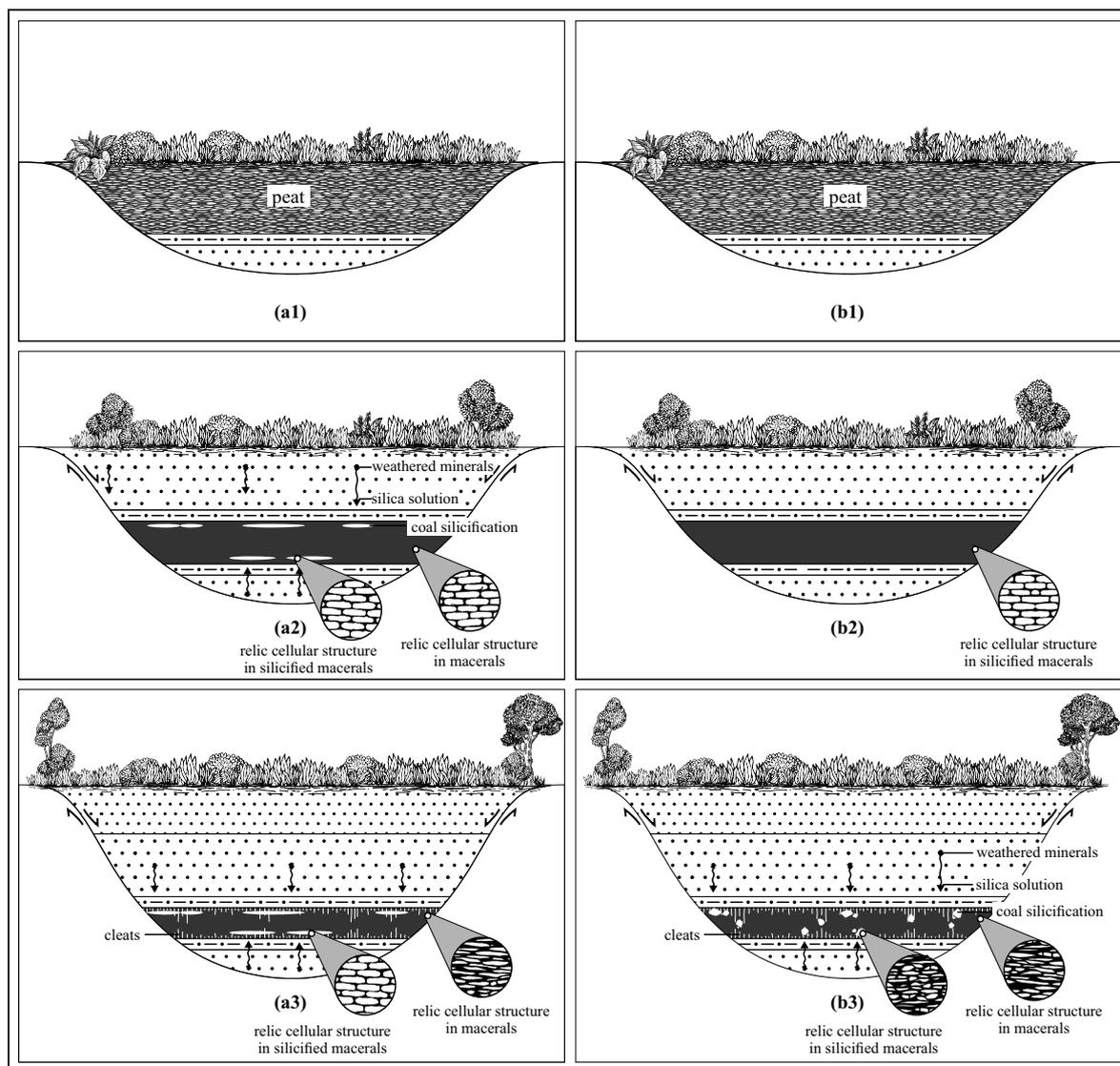


Figure 7. Illustration of reconstructed silicification models in the layered (a) and nodule (b) types.

became compacted, but the silicified areas would have remained uncompacted as noted by suberinite maceral observed in both coal and silicified coal samples (Figures 7b, d, f, h). In Figure 7b3, the underlying and overlying rocks were likely altered in this later coalification stage, promoting silica dissolution and thus silicifications in the coal seams. The permeation of silica solution in coal seams might be limited through cleats as the coal matrix became less porous. A smaller reactive surface area in the less porous coal allowed silica solution to penetrate across the coal seams. In such cases, the mineralization sites would have been controlled primarily by the cleat network

within the coal seams. As the mineralization took place during later diagenesis where the organic matter has been degraded, macroscopic layered and preserved cellular microstructure are rarely seen on the silicified coals.

### CONCLUSIONS

Geochemical and petrographic analyses have demonstrated the genesis of coal silicifications in samples from Tanjung Redeb, Embalut, and Loa Kulu. The silicification process was most likely initiated by the weathering (dissolution) of

rock forming minerals present in the underlying and overlying sedimentary rocks. The solution would, therefore, permeate to the coal seams and precipitate silica minerals. Layered silicification seems to occur during relatively early diagenesis through replacement and permineralization, leading to anatomical structure preservation of the precursor tissues. Nodule silicification likely took place during later diagenesis when the organic matter had been compacted and more physically and chemically degraded. Mineralization in this circumstance would not preserve the anatomical structure. It appears that the silicification tends to occur within huminite rather than inertinite and liptinite macerals. Coexistence of silica polymorphs consisting of opal, chalcedony, and quartz is noted in the Tanjung Redeb silicified coal samples. Chalcedony showing fibrous internal structure and botryoid surface structure may indicate transformation from opal during diagenesis.

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