

On the silica diagenesis processes of the coal debris from Miocene formation from Prahova County

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Belonging to the category of gems, in the same division with amber, silicolitic insertions or agates, silica wood are quasi-present in Oligocene and Miocene age arenitic or coal lithofacies as well as in volcanogenic-sedimentary formations with post-geyser activity, or as remaking in the Salt Breccia.

The speciality literature mentions frequent occurrences of silica wood in Oligocene and Miocene sand and coal formations, from the Transylvanian basin, Eastern Carpathians, Southern Carpathians and Western Carpathians.

Known under the name of xiloid agates, silica woods can be gemologically attributed to jaspers and according to their degree of the replacement of wood tissue with silica; they may form species aesthetically comparable with agates. Silica within coal wood appears under the form of chalcedony or, sometimes opal-like.

In time due to the fact that they lose water, these types of silica change to quartz. The good fossilization reveals minute details and the xilotomic identifications associated with palynological data may allow paleofloral diagnosis.

In the geological deposits two main types of silica wood are cited: 1. the ones which were partially carbonized and afterwards silicified (they have dark colours, even black); 2. the ones which were just silicified (they have light colours, even bright ones). The former ones are connected to coal-clays formations, the latter appear in sandy volcanogenic-sedimentary formations or geysers ones.

In comparison with famous occurrences, silica woods with big dimensions and varied colours (brown-grey, red, yellow, black, policolour) such as the stone forests from Red Deer Calley-Alberta, Canada, Wyoming Valley, Virgin Valley – Nevada, the national park Holbrook – Arizona, USA, Daskel Moka Ham – Egypt, Bulgaria, Argentina, Madagascar, Hoggar Mountains – Sahara, in Romania reduced formations were found, they represent reworked sporadic fragments of tree trunks. We don't have big accumulations in situ registered.

The most frequent occurrences are those from sandy, siliceous and coal lithofacies of Oligocene and Miocene formations from the Transylvanian basin (reworked fragments of trunks in alluvial deposits of Almaș Valley, Agriș Valley, Gârbului Valley, Cristolțel Valley) or Eastern Carpathians (the lithofacies of Kliwa silica sandstones with levels of dysodilic shales to which we may add silicolithic formations of lower or upper menilites). We may add silica and coal wood in Oligocene formations from Petroșani Basin.

The samples of silica wood, previously coal wood have dark colours (grey and black shadows) and the main mineralogical element for this type is chalcedony.

In the collection of the section of petroleum geology from Petroleum-Gas University of Ploiești there are a few fragments of metrical trunk silica wood previously coal wood, prelevated from the dysodilic levels of the formations of upper Kliwa sandstone, from the canyon dug in Zâmbroaia. More samples of small dimensions come from dysodilic levels of Kliwa sandstone, from Sibiciului Valley; they were also found

south to crop with Kliwa sandstone, in the alluvial deposits of Zâmbroaia, Matița and Slănic of Buzău Valley, within the area of Miocene salt breccia.

Data from literature (Petrescu I et al., 1989) refer to silica wood from lutitic levels of Kliwa sandstone at taxodiaceae (*Sequoioxylon gypsaceum*), lauracea (*Laurinoxylon murgoci*) and *Icacinoxylon species*. The palinological frame of lutites from Kliwa sandstone is dominated by conifers (40-52%) and angiosperms (45-55%), with pollen of *Cupuliferoidae polenites*, *Liblarenzis* and *Ulmipolenites undulosus* type prevailing.

On the coal and silica wood from Zâmbroaia geologists have made investigations of optical and electronic microscopy, whose analysis is made up as pretext of several theoretical considerations which as integrated within the geologic frame would allow the reconstitution of silica processes.

The upper Kliwa sandstone is developed along Zâmbroaia Valley, making the flanks of Alunișu syncline and of Zâmbroaia anticline, on which it imposes a relief, specific to canyons. The maximum thickness of the formation is 600 m, and within it there may be distinguished three piles gradually and laterally passing from one to another. At the bottom, there is an alternation of thin strata of dysodilic shales and subordinated Kliwa sandstones. In the dysodilic intercalations from the north flank of Zâmbroaia anticline several trunks with the length of about one meter and a diameter of 20-30 cm of carbonified wood at surface and silicified ones inside. The medium pile contains an alternation of Kliwa sandstone which are thicker and dysodilic and dysodiliform shales.

The upper pile contains massive metric banks of Kliwa sandstone alternating with thin subordinated levels of dysodilic shales. As levels, there are siliceous stones of gaize type, brown diatomites in fresh broken fragment, white or yellow by weathering; at the bottom, the well-cemented grey, siliceous sandstones, micaceous of Fusaru type which contrasts under the sandy uncemented aspect of Kliwa sandstone. Sometimes, there are diatomites and rare thin microbreccia levels with elements of green schistes. From the micrographic point of view, the mineral components of Kliwa sandstones are made up of about 98% quartz, with very little feldspars and 0,2% heavy minerals (ilmenite, kyanite, zircon, staurolite, tourmaline, rutile, garnets, sphene) and micas (muscovite, biotite, chlorite), typical especially to the metamorphic stones than the igneous ones.

The clasts are very fine, well-sorted, frequently angular, immature, transparent (medium-maximum) and about 10% are round, corodated, with little scales and little holes of aeolian impact. We may add secondary minerals (glauconite, pyrite, iron oxides) and very rare bioclastes (spicules of sponges, tissues of radiolars, diatomea). The cement is frequent of opal + chalcedony, opal + chalcedony + secondary quartz, when it comes from siliceous organisms and sometimes, it is mainly calcitic or the change of the opal with calcite. In comparison with the lower Kliwa, in upper Kliwa sandstone almost all the bioclastes lack, the glauconite is more frequent, the pyrite crystals are more developed, and the thickness of the banks is greater.

Structurally speaking, Zâmbroaia anticline is normal, right, asymmetrical with the north flank more developed, made up of Oligocene and Miocene deposits in depth. It is crossed by Zâmbroaia Valley on a length of 2000 m. From Păcuri peak to the east it is affected by an axial fault along which the south flank is sunk. Its amplitude increases to the east in the sense of plunge to the east and to the south it is covered by the

transgressive Meotian, under which the Miocene screen qualities are developed, giving importance for hydrocarbons to the east of Păcuri peak. To the east of Zâmbroaia Valley, Zâmbroaia anticline is sunk in transversal faults in steps to Valea Gardului, being covered by the Miocene of Predeal Sărari syncline.

The attempt to discover the way of appearance of silica wood presupposes several theoretical considerations referring to the silica diagenesis. We will follow control factors of silica solubility and general diagenesis mechanisms will result.

The field and laboratory observations suggest that generalized silica diagenetic sequence follow the scheme: opal-A (biogene and siliceous oozes)→opal-A' (amorphous nonbiogene silica)→opal-CT→opal-CT-more ordered-cryptocrystalline and chalcedonic quartz→microcrystalline quartz. The changes: opal-A→opal-CT→quartz is achieved on dissolution-reprecipitation reactions.

In systems of low temperature, silica precipitates from natural water solutions such as the amorphous silica (opal-A). The precipitation is made up by means of organisms (diatomea, radiolars) whose tissues accommodate in siliceous oozes. The progressive diagenesis with depth presupposes the fragmentation of tests (fig. 1, *a*), partial dissolution and precipitation of silica of over increase (fig. 1, *b*) which sometimes may be opal-A'. Both opal-A and opal-A' are amorphous on the diffractograms RX, with large, diffused, central peaks around the value of 4.1Å (fig. 2, *a, b*).

At a bigger burial depth, the polymorphic amorphous silica is in a disordered phase, less soluble of cristobalite – tridymite (opal-CT) (fig. 1, *c*). At the analysis of RX diffraction, opal-CT reveals a decrease of spacing $d[101]$ from 4.1Å to 4.04Å, as well as an increase in the relative order of cristobalite in comparison with tridymite. Opal-CT recrystallizes to the quartz less soluble phase (fig. 1, *d, e, f*). The quartz crystallinity increases progressively with the crystal growth, eventually equalizing the one of igneous and metamorphic quartz crystals (fig. 2, *e, f*).

The changes follow the stage of dissolution and successive reprecipitations. Thus, the organic tissues are fragmented, dissolute and reprecipitate as opal A' and then as opal-CT. Opal-CT is deposited within the tissues, within the space of pores or the liberian-wood capillars as cements, lepispheres (sferoids of 1-10 μm) and blades of a thickness of 10-100 nm.)

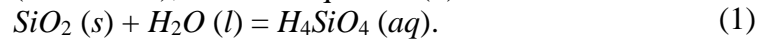
At a scale of about 10 nm, structures change in a manner of transformation which is apparently in a solid form. Therefore the fabric of sediments is kept, and the lepispheres which fill the pores may appear as pseudomorphosed in quartz cherts. The preservation of the sediment fabric which we noticed suggested even the change in solid form opal-CT→ quartz. Actually, preserving the fabric presupposes a mechanism of grain-to-grain substitution with low dissolution and local precipitation rates.

Chemically speaking, the diagenetic sequence is thermodynamically and kinetically checked. Ideally speaking, at a certain moment, in the shallow crust only the most steady silica polymorphs can be present (this would be the quartz phase). Actually, the change rates between polymorphs are low, and the metastable polymorphs, once having been precipitated, may persist (we may cite forms of opal-A or opal-CT from Cretacic or older formations). The simple transformations of the silica diagenetic sequence: opal-A, opal-CT, quartz are due especially to the relation between the

solubility of a polymorph and the shallow area or the particle dimension (implicitly the free surface energy).

The temperature subordinately influences the changes (at most it can accelerate reactions, shortening the phase change time).

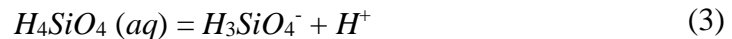
From this point of view, silica is dissolved in solution of $pH < 9$ in order to produce undissociated H_4SiO_4 (silicic acid), as in the equation (1):



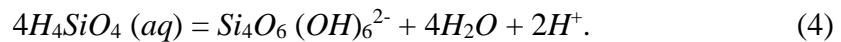
The equilibrium constant presupposing the unitary activities of solid and water silica is:

$$K = a_{H_4SiO_4}. \quad (2)$$

If the system is in equilibrium, K is a real thermodynamic equilibrium constant, or product of solubility K_{sp} ; if the system is not in equilibrium, K is an ionic activity product, IAP . The quantity $a_{H_4SiO_4}$ is the thermodynamic activity of H_4SiO_4 , undissociated (monomeric). Each silica polymorphous has a characteristic K constant. The solubility of silica polymorphs equalizes the concentration of dissolved total silica. Neglecting the differences between activity $a_{H_4SiO_4}$ and concentration $[H_4SiO_4]$ (presupposing that they are equal), the solubility is greater than $a_{H_4SiO_4}$ for which the silica dissociation extension, the complexation, polymerization and hydrolysis appear. In a system rich in silica at $pH > 9$, we can speak about the appearance of silicic acid dissociation and its polymerization in the reaction:



and



$H_3SiO_4^-$ and $Si_4O_6 (OH)_6^{2-}$ contribute to the total dissolved silica ($\Sigma SiO_2 (aq)$), and therefore:

$$\Sigma SiO_2 (aq) = [H_4SiO_4] (aq) + [H_3SiO_4^-] (aq) + 4[Si_4O_6 (OH)_6^{2-}] (aq) + \dots \quad (5)$$

Therefore, $\Sigma SiO_2 (aq)$ is dependent on $pH > 9$.

The oversaturation degree (Ω), defined as:

$$\Omega = \frac{IAP}{K_{sp}} = a_{H_4SiO_4}. \quad (6)$$

If $\Omega > 1$, reprecipitation may appear and if $\Omega < 1$ dissolution may appear. Equation 6 demonstrates that $a_{H_4SiO_4}$ and not $\Sigma SiO_2 (aq)$ is the one defining the oversaturation.

The effective size of K_{sp} depends on the type of polymorphs and on its dimension and its form. The size $a_{H_4SiO_4}$ depends that $\Sigma SiO_2(aq)$, on pH and the solution composition.

In nature the more porous tests or tests with an irregular surface are preferentially dissolved, therefore leading to the local reprecipitation of the less soluble phase of opal-A' (precipitated secondary opal-A' is cited in postdepositional stages of the early diagenesis). Part of opal-A' may be opal-CT as the first stages of the transition from opal-A at opal-CT precipitation. After these crystalline inclusions we gradually start the increase of greater, blade-like lepispheres of opal-CT. In time, these ones beget lump morphology (fig. 1, c) the increase of opal-CT crystallinity (fig. 2, c, d) may appear due to the increase of crystal dimension and the preference of cristobalite to the tridymite in the maturation process.

The first quartz which precipitates has reduced grain dimensions and shows thin crystallinity in RX diffractograms (fig. 2, e). After precipitation, the crystal dimensions and the quartz crystallinity increase.

In an impure system, the complexing and the adsorption may induce variations in the reaction rates: 1) opal-A changes directly to quartz; 2) the clay formation or the formation of zeolites on account of the opaline silica; 3) variations in the silica change rates due to impurities (the change delay or acceleration due to detritic minerals or autigene formation in the relation solubility the specific surface area).

In conclusion, the silica diagenetic evolution is controlled by the solubility in water of phases (polymorphs) which is function of the crystal structure and the particle dimensions and form.

The silica concentration in sediment water pores depends not only on the morphology and crystalline structure but also on the water pH, temperature, pressure, type and concentration of other silica dissolved species (water complex polymers), additional minerals from the sediment.

The diffusion rates of pore water (controlled by the sedimentary fabric) are important as well in the sense that large diffusion rates prevent the *in situ* concentration which is necessary to a given precipitation.

The basic pH increases the silica solubility in the sense that it facilitates the ionization of monomeric silicic acid H_4SiO_4 , predominant in unsaturated solutions.

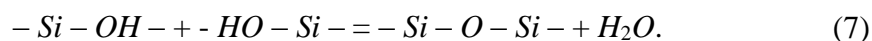
The temperature increases the silica form solubility and the diagenetic rate reactions (fig. 3).

The silica polymerization takes place in solutions which became oversaturated as a result of a net pH fall, temperature or fluid volume (by evaporation).

Observations and experiments described in literature created a silica diagenesis model. The silica polymerization is produced for both oligomers (dimers, tetramers) and species with a 10^4 degree molecular weight and 1-5 mm diameters. The silica polymer growth is done by maturization (less big particles increasing on account of more little particles). The continuation of maturization allows the appearance of colloidal dimension particles which either remain in the suspension as soils (at basic pH and reduced salinity) or coagulates and gellifies (at acid pH or higher salinity) (fig. 4).

Oligomers (dimers, tetramers) exist at low concentrations and even in subsaturated solutions at lower temperatures than 130°C, and at higher temperatures, saturated solutions in quartz seem to be predominantly monomeric.

Within polymerization, smaller oligomers form ring structures which condensate groups at silanol ($-Si-OH-$) to produce siloxane ($-Si-O-Si-$) of bigger polymers as in the reaction:



Due to these external silanol groups, the silica polymers have a negative residual surface load. The zero load point (meaning the pH point at which there is no residual surface load on the molecule) is very reduced for the amorphous silica (2 ± 0.5) and represents a major control of polymeric and colloidal silica deposition.

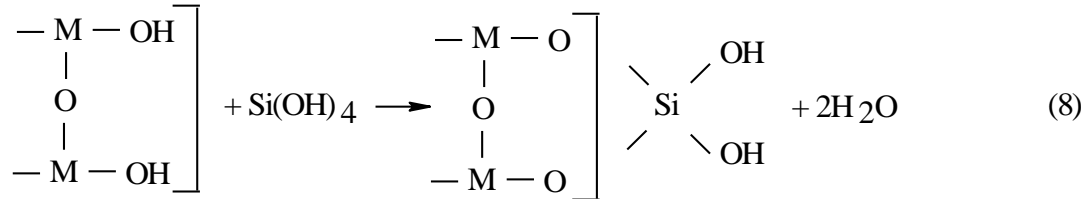
The silica colloids and silica substrata reject each other, even if their surface loads are not bridged or neutralized by counter ions such as the polyvalent cations. The effects of salts on silica colloids are illustrated in figure 4.

In oversaturated solutions three different polymerization mechanisms operate:

1) Polymers of colloidal dimensions may flocculate in the presence of cations. Alternatively less active polymers may depolymerize evolving to other mechanisms (2 and 3).

2) The molecular deposition of monomeric and oligomeric silica may be catalyzed by hydroxyl ions (fig. 5).

The deposition appears as condensation reactions on SiOH or MOH surfaces as in reaction 8:



In this case, one the first silica monostratum is deposited, this one becomes a reactive substratum. The silica may be removed from water by means of coprecipitation with insoluble metal hydroxides, for example $Mg(OH)_2$. In substaturated solutions the silica may coprecipitate with Zn, Al, Fe, Mn, Mg hydroxides.

3) The lower polymer depolymerization represents a continual source of monomeric silica. The oligomers fed from silica sols may depolymerize as monomers which condensate on bigger colloidal particles in increase. This overshooting may be attributed to situation when the dissolution rates are bigger than deposition rates.

We should also mention the influence of the adsorption behavior as result of the polymer negative shallow load effect. The colloidal silica sols precipitate through polymerization in an oversaturated alcanic solution of low salinity for opal-A.

The biogenic opal-A is extracted from oversaturated solutions through the catalytical enzymes. On anorganic account, the oversaturation may be reached through pH fall, temperature decrease or evaporation. Opal-A in solid form is deposited with sediments where particle surface irregularities dissolute and reprecipitate secondary opaline fabrics with a more ordered structure.

If the silica is dissolved in waters with salt pores open frame polymers are formed. Through their stocking six opal-CT member ring structures result (format which is preferred to quartz).

The clays within the system adsorption silica delaying the deposition process. In the presence of carbonates in dissolution, the formation of positively loaded hydroxyl $Mg(OH)_2$ type complexes may flocculate opal-CT.

After opal-CT deposition, the crystallinity progressively increases on account of the preferential dissolution of tridymite strata and cristobalite strata precipitation.

When in the opal-CT reordering, d[101] spacing is about 4.04Å opal-CT is redissolved. Clays and/or carbonates, along with the burial temperature increase favor a reduced level of pore water saturation, creating conditions for the slow quartz precipitation in a monomeric solution. In time quartz crystals increase their dimensions and get the well ordered structure of the ones in igneous and metamorphic rocks.

In the photos of optical microscopy on thin sections liberian-wood capillars filled with lepispheres appear, as well as joints filled with lepispheres.

In the photos of electronic microscopy 5000 and 10000 x bigger euhedral quartz crystals appear at the contact with an amorphous entity in the rest of the photos an amorphous entity can be noticed.

According to F.K. Dresche-Kaden (1969) the silica source in the wood fossil silica processes would be its liberation from the surrounding minerals and clays due to humic acids from coal lithofacies.

According to H. Erhart (1973), the humic acids brought by the continental soils in the sedimentary basins could play the leading role in the wood fossil silica processes. He claims that the silica source is predominantly the phytolithe depolymerization, corpuscles from continental plant walls. The period of time was initially used to rename the fine tissue or the colloidal silica corpuscles accumulated in the plant walls as hormonal products of their metabolism. These particles having the dimension of 5-10 μm are made up of opal and when the plant dies it accommodates in the soil, subsequently being washed by rivers and transported in sedimentary basins. This silica of organic origin is more soluble than the one in silica processes and allowed an easy impregnation of the wood tissue.

In the selective silicification of fossil woods to the context in which they appear silica solutions appeal to the attraction of organic matter. The different porosity, the presence of vacuum cells, the concentration difference of cellular solution, the osmotic predisposition, the humus presence or the subsequent phytolithe within their wood structure would explain the silicification selectivity.

We may add to these suppositions the fact that the pH and temperature differences, and therefore silica solubility and subsequently saturation, at the passing of arenitic lithofacies of Kliwa sandstones into the anoxic dysodilic lutitic lithofacies, enriched with remanent organic substance favor the silica precipitation.

The presence of fossil woods confirms the proximal shelf ambience in which Kliwa arenites were deposited. The impermeable kind of the lutitic context of disodiles facilitated the carbonification allowing the subsequent silicification.

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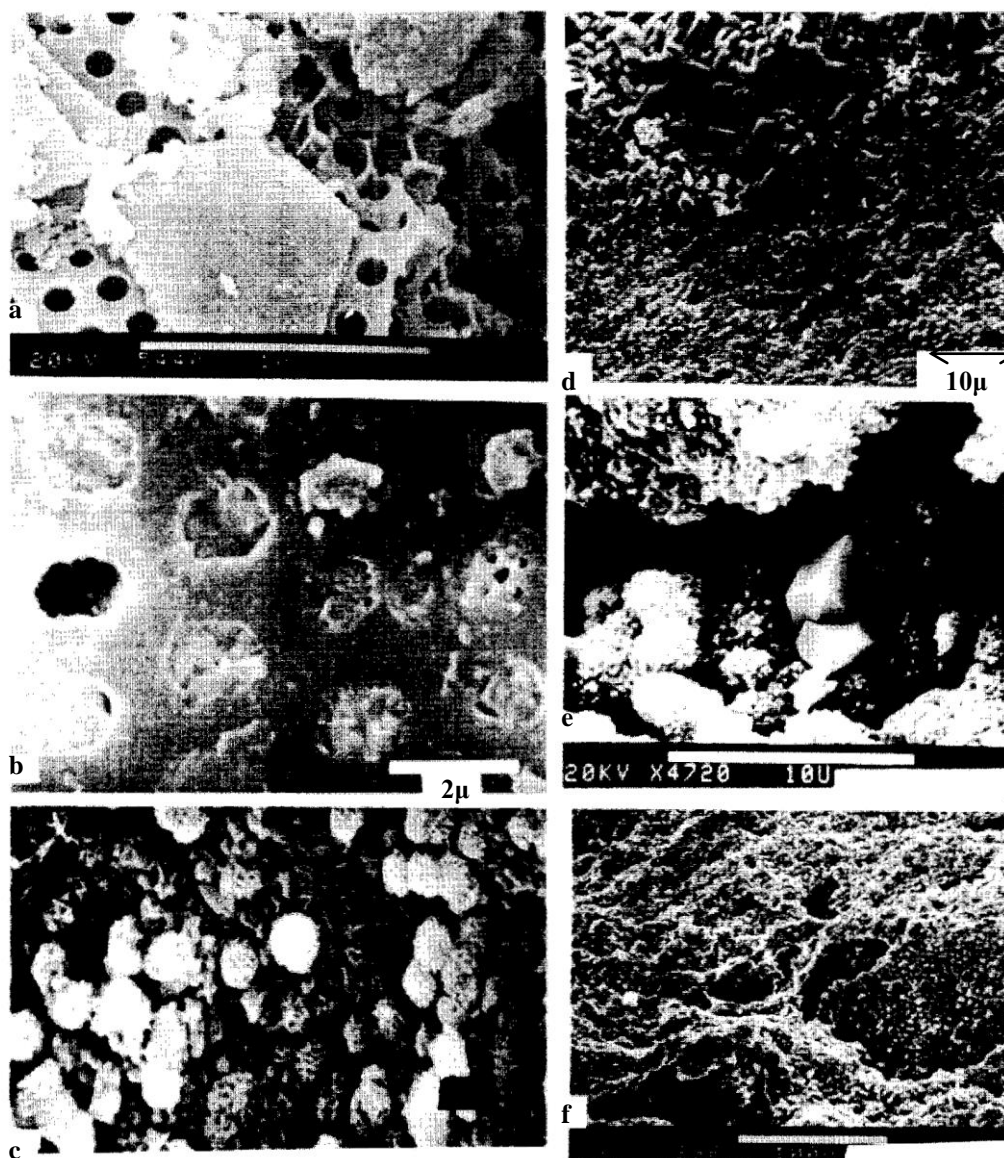


Fig. 1. – a) Siliceous microfossil ooze, in this case a diatomite from the upper Miocene Monterey Formation, San Joaquin Valley, California. b) Pristine diatom frustules and an enlargement of a broken frustules in which opal-A' is being deposited as a pore-filling. From Hein and others (1978). c) Opal-CT lepispheres, in this case forming as a replacement of a diatom frustules. Monterey Formation (Miocene), San Joaquin Valley, California. d) Abrupt contact between opal-CT (upper portion) and quartz (lower portion) in a nodule. Note immediate change in texture, crystal size, and porosity across boundary. Monterey Formation (Miocene). From Grivetti (1982). e) Euhedral quartz crystals forming in a vug in an opal-CT rock. Monterey Formation (Miocene), San Joaquin Valley, California. f) Monterey Formation sample (Miocene) which has been totally transformed to poorly crystalline quartz. Recrystallized diatom indicates the biogenic origin of the rock. San Joaquin Valley, California.

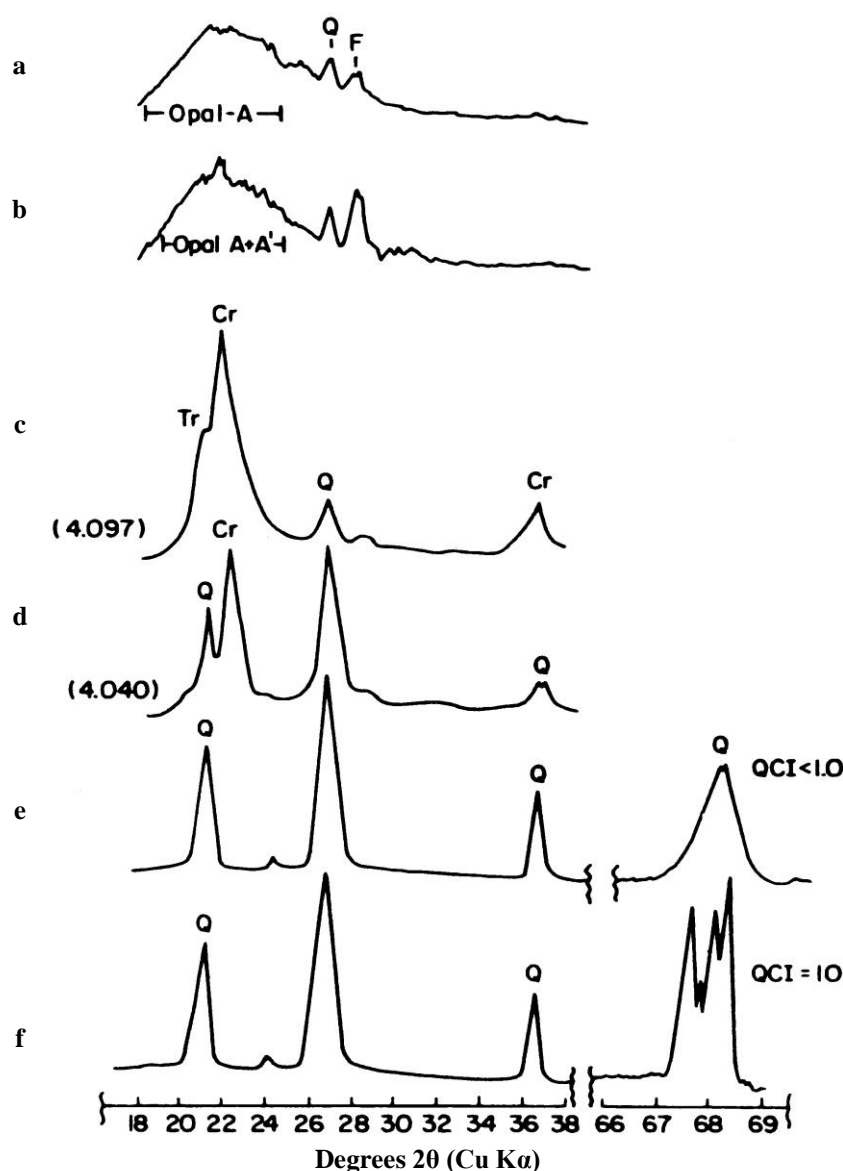


Fig. 2. – XRD patterns representing the diagenetic sequence. Q = Quartz (either detrital, as in a-c, or diagenetic, as in d-f). F = Feldspar (detrital). Cr = Cristobalite. Tr = Tridymite. a and b modified from Hein and others (1978); c-f modified from Murata and Larsen (1975) and Murata and Norman (1976). a) Diffuse hump between approximately 19° and 25° 2θ representing opal-A. b) Opal-A hump showing development of small peaks in the 19° - 25° 2θ range, reflecting early precipitation of opal-A'. c) XRD pattern for opal-CT, with $d[101]$ -spacing = 4.097\AA . d) XRD pattern for opal CT showing increasing crystallinity and decreasing d -spacing with depth. $d[101]$ -spacing = 4.040\AA . e) Transformation of biogenic silica from opal-CT to poorly crystalline quartz. Quartz crystallinity index, measured from the (212) peak of quartz at 67.76° 2θ , = < 1.0 . f) Increased diagenesis causes authigenic quartz to become better ordered, as denoted by a well-formed quartz (212) peak at 67.74° 2θ . Quartz crystallinity index of this sample = 10.0 .

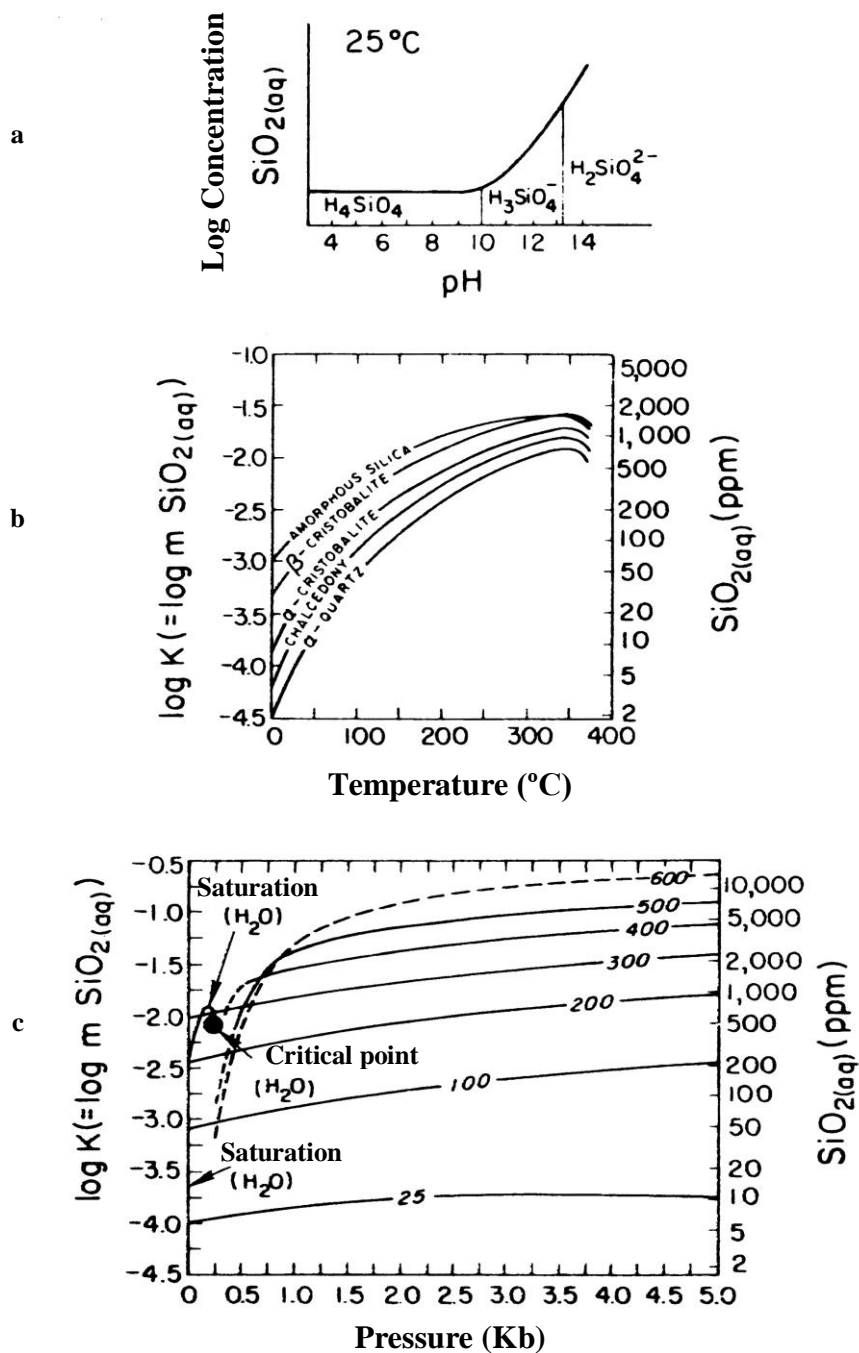


Fig. 3. – a) Increase in silica solubility with increasing pH (after Volosov et.al. 1972). b) Increase in silica solubility with increasing temperature for several silica polymorphs (modified from Walther and Helgeson 1977). c) Increase in quartz solubility with increasing pressure at varying temperatures (modified from Walther and Helgeson 1977).

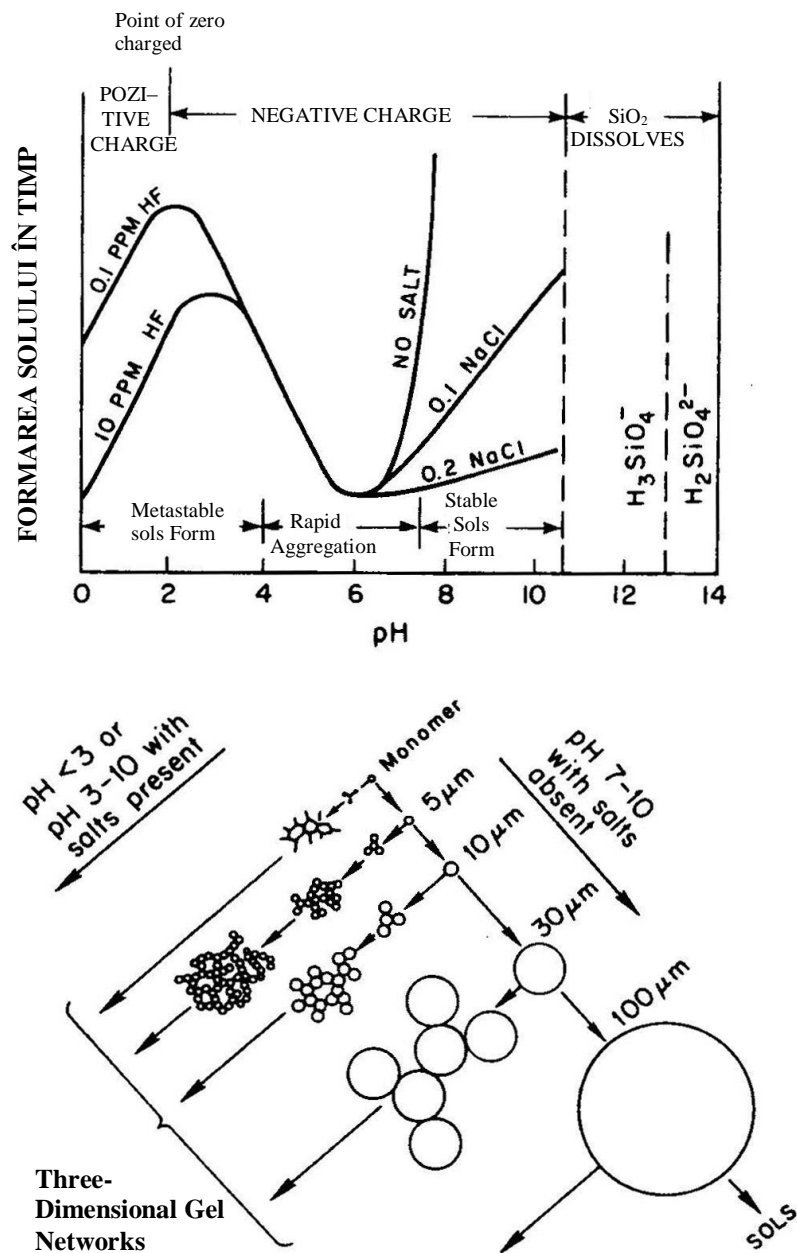


Fig. 4. – a) Effect of pH on the colloidal silica-water system. Below the point of zero charge, silica particles are positively charged, while above this point, silica particles are negatively charged. At low pH, metastable solids form due to trace amounts of fluorine in the systems. Stable sols form primarily in the pH range of 6.5-10. At higher pH, silica dissolves, and sols will not form. Simple salts, such as NaCl, will retard sol formation (after Iler 1979). b) Polymerization behavior of silica, in solutions saturated with respect to amorphous silica. In basic solution, with low salt concentration, particles grow in size while decreasing in number (i.e., opal-A precipitation). In acid solution, or in basic solution with high salt concentration, particles aggregate into three-dimensional networks and form gels (i.e., opal-CT precipitation) (after Iler 1973).

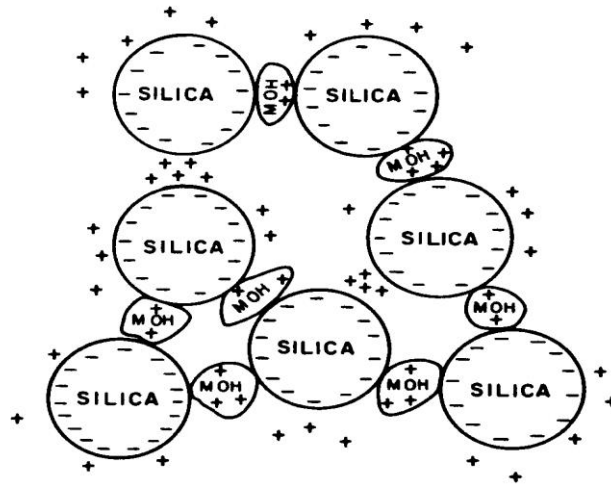


Fig. 5. – Silica particles, which have a negative residual surface charge, can be flocculated by formation of positively charged hydroxyl complexes (such as $Mg(OH)_2$) (after Iler 1979).

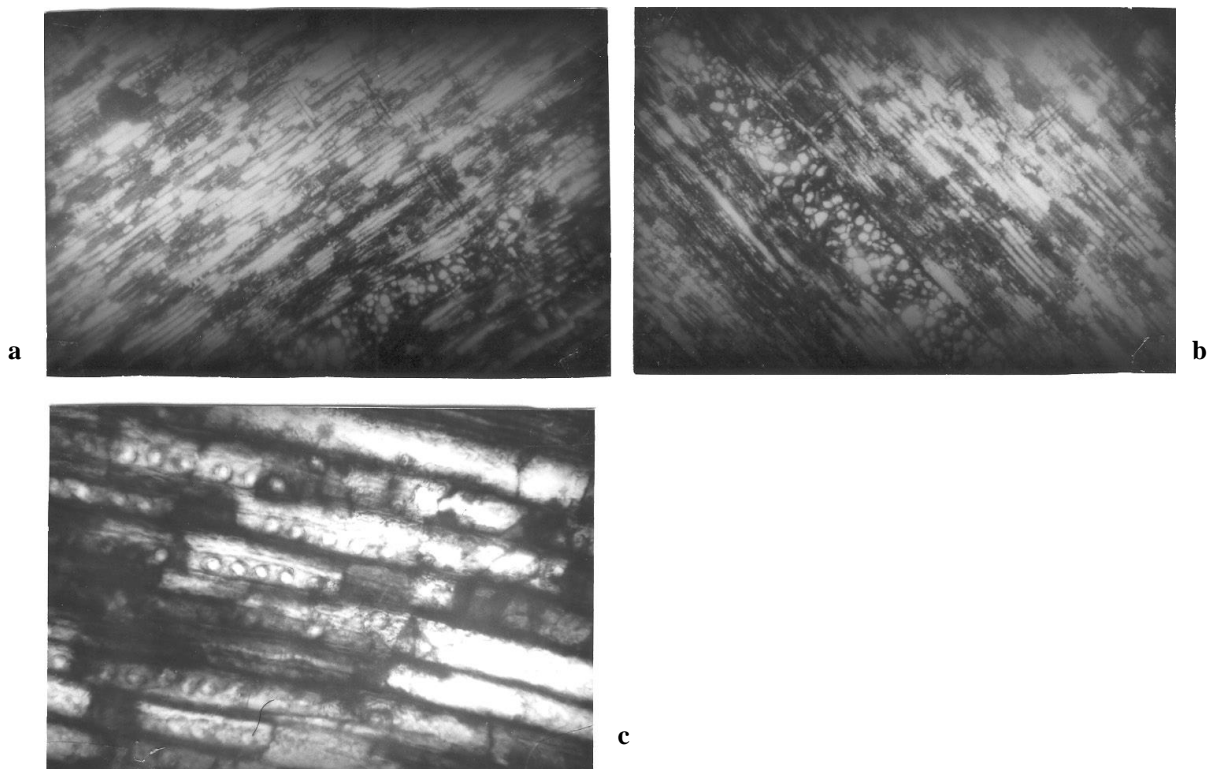


Fig. 6. Thin sections in coal silicified wood from Zâmbroaia Valley. Note liberian-wood capillars filled with amorphous silica lepispheres. crossed nicols, increase: a, b – 75 x; c – 150 x.

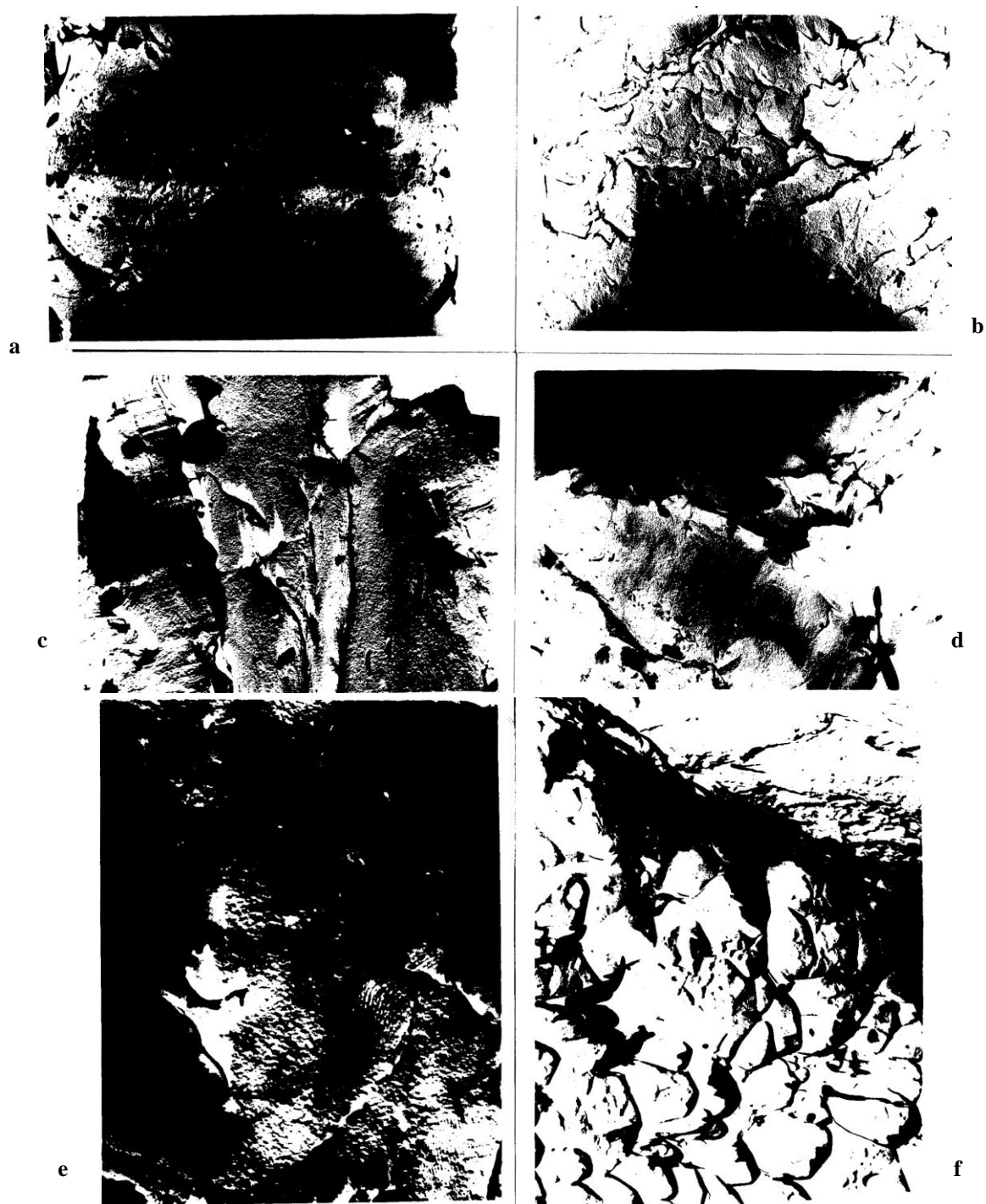


Fig. 7. Photos at SEM in silicified coal wood from Zâmbroaia Valley. a – e: amorphous silica (5000 x increase); f – abrupt contact marked by coal tissue which separate amorphous silica from quartz subhedrale crystals (10000 x increase).