CONTRIBUTIONS OF FOREST OPAL AND ASSOCIATED CRYSTALLINE PHASES TO FINE SILT AND CLAY FRACTIONS OF SOILS

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Abstract-The scanning eiectron microscope (SEM) is useful in the identification of biogenic opal. Opaline spheres, cups, and scrolled or convoluted sheets were identified in both soil and vegetative isolates. X-ray diffraction analysis indicates that both alpha quartz and cristobalite were co-associated with the amorphous opaline phase synthesized during life metabolism of deciduous tree leaves. Such crystalline phases were most abundant in the $2-5 \mu m$ fraction and many consist of anitsotropic rods with parallel extinction or equidimensional bodies with aggregate extinction. Between $2/3$ and $3/4$ of the total opal isolate from deciduous tree leaves was solubilized when digested for 2.5-min in boiling 0.5 N NaOH. Rate of dissolution was a function of particle-size and tree species. Biogenic opal of forest origin was about 10-15 times more soluble than grass opal, which probably reflects the higher specific surface of the former.

silicate with a formula of $SiO_2 \nvert A_2O$. However, more mura and Kanno, 1965; Wilding and Drees, 1968, refined definitions indicate its composition includes 1971, 1973; Wilding *et al.,* 1967; Twiss *el ai., 1969;* submicroscopic crystallites of cristobalite, α quartz, or Rovner, 1971; Bonnett, 1972; Geis, 1973). trydimite in addition to the X-ray or optically amor- To date, little is known concerning the ultimate fate

(organic) origin occur as residue products in soils and as 50 to 75 per cent of the total opal contributed by geologic deposits. In general, opal of plant origin is plants is reported to be in \lt 5 μ m fractions (Jones and optically isotropic; its specific gravity ranges from 1,5 Beavers, 1964; Wilding and Drees, 1971; Geis, 1973). to 2.3, refractive index from 1.41 to 1.48 and transmit-
Wilding (1967) has shown that under certain soil conflect higher quantities of occluded organic carbon pig- for at least 13,000 yr. Opalized bodies have been isomentation. In $> 5 \mu$ m fractions, opal conforms closely lated from geological sediments of Tertiary age (Gill, to the cell in which it originates, unless subsequently 1967), The presence of free iron and aluminum oxides fragmented. Scanning electron micrographs indicate under acid forest soil environments should favor the that opaline constituents of forest origin are generally stability of opal. Lewin (1961) and Jones and Handreck thin $(1-2 \mu m)$ fragile plate-shaped incrustations of cell (1963) have found evidence that Fe and Al are chemiswalls and seldom form solid polyhedral structures orbed to opaline surfaces, thereby retarding subcharacteristic of grass opal. The size of opal varies con- sequent silica dissolution. Further, it has been shown siderably among anatomical components within a spe- that opaline constituents undergo partial or full confrom about 1 mm to $\langle 2 \mu \text{m}$. More comprehensive sediments (Beavers and Stephen, 1958; Yarilova, 1952; reviews of optical, morphological, physical, and chemi- Mizutani, 1967). Several workers have presented X-ray cal properties of biogenic opal may be found elsewhere evidence of cristobalite, trydimite, or quartz in opaline (Smithson, 1958; Beavers and Stephen, 1958; Siever isolates of vegetative tissues but generally attributed

INTRODUCTION and Scott, 1963; McKeague and Cline, 1963; Jones Opal is commonly considered a hydrated amorphous and Beavers, 1963; Jones and Handreck, 1967; Ari-

phous silica phase (Teodorovich, 1961; Jones *et al.*, (rate of dissolution and stability) of biogenic opal in 1963,1964). Evidence from this work suggests that every soils, particularly under forested conditions. Little intermediate stage between amorphous silica and attention has been given to opal characteristics. transquartz exists. The contract of the correct of the formations, or co-associated crystalline phases in fine Many forms of opaline constituents of biogenic silt $(2-5 \mu m)$ or clay $(< 2 \mu m)$ fractions. Yet, as much ted color from colorless to opaque. Darker forms re-
ditions grass opal in the $50-20 \mu m$ fraction may persist cies and from species to species, but commonly ranges version to chalcedony upon aging in soils and geologic such crystalline phases to artifacts produced by dryashing isolation procedures (Lanning *et al., 1958;* Jones and Milne, 1963; Arimura and Kanno, 1965).

The objectives of this work were: (a) To identify characteristic forms of opal concentrated in the $\lt 5 \mu m$ fraction of reference deciduous tree-leaf isolates; (b) To determine if characteristic forms of opal persist and can be identified in the $\lt 5 \mu m$ fraction of forest soils; (c) To determine what crystalline silica phases are coassociated with opaline isolates from forest soils and reference tree leaves; and (d) To determine the alkali solubility of opaline isolates from reference tree leaves.

METHODS AND MATERIALS

Opaline constituents were quantitatively isolated from the $2-5 \mu m$ fraction of surface (AI) horizons of three forested soils (Miamian series) using sink-float heavy liquid separations similar to those outlined by Jones and Beavers (1964). The heavy liquid was a solution of nitrobenzene-bromoform with a s.g. of 2.30 . A 9 percent polyvinylpyrrolidone solution in ethanol was employed to facilitate dispersion similar to the method described by Henderson *et al.* (1972). Total clay $(< 2 \mu m$) of the above soils was also examined for opal with a scanning electron microscope. No opal yield could be obtained from clay fractions using above methods.

Reference opal isolates from deciduous tree leaves common to the forest soil sites were obtained using methods previously reported (Wilding and Drees, 1971). Senescent leaves from the following trees were examined: American beech *(Fagus grandifolia),* sugar maple (Acer saccharum), white ash (Fraxinous ameri*cana),* white oak *(Quercus alha),* American linden *(TUa americana)* and hackberry *(Celtis accidentalis).* Prior to opal isolation the leaves were carefully scrubbed in distilled water to remove any possible adventitious contaminants and later checked under a microscope to confirm their absence. Two methods were employed for opal isolation: (a) combined dry-ashing (300- 400°C) followed by wet-ashing in concentrated H_2SO_4 ; and (b) low-temperature ashing (60–65°C) employing a Coleman Model 40 R-F reactor with an oxygen atmosphere. The latter method was a check to see if crystalline artifacts were produced by ashing specimens at $300-400^{\circ}$ C. Opaline isolates were then fractionated into $2-5 \mu m$ and $\langle 2 \mu m \rangle$ separates employing standard sedimentation methods.

Specimens of $2-5 \mu m$ opal were then affixed to a standard microscope slide with epoxy resin and coated with about 200 Å of gold. They were prepared in such a manner that mounts were suitable for both light optical and scanning electron microscopy (SEM) observations (Wilding and Geissinger. 1973). Clays, suspended in acetone, were plated on a glass slide prior to coating. Additional details concerning operational parameters of the JSM-U3 SEM can be obtained from the latter report.

For X-ray diffraction analysis a Norelco unit equipped with a Cu-target X-ray tube, a Ni K - β filter and a proportional counter was employed. Linear scans from 14° to 34° 2 θ at a rate of 2° 2 θ /min were made with a I-sec time constant, 0·006 in. receiving slits. A 10-V base line and 8-V window were set on the pulse height analyzer.

The $5-20 \mu m$ and total opal isolates of American beech, white oak and sugar maple were given an alkali dissolution treatment consisting of a 2·5 min digestion in boiling 0·5 N NaOH (Hashimoto and Jackson, 1960). Weight loss after this treatment was determined. Residues were subsequently examined optically, with SEM, and with powder camera diffractometry.

RESULTS AND DISCUSSION

Opaline morphology

Characteristic spheres, cups, rods, stomata guard cells, scrolled sheets and tabular mosaic aggregates or bladed bodies can be identified in the $<$ 5 μ m opal isolates (Figs. 1–6). Similar forms isolated from $> 5 \mu m$ fractions of forest vegetation and soils have been reported previously (Wilding and Drees, 1971, 1973).

Spheres, perhaps one of the most distinctive of the above forms, are frequently attached to host structures (Figs. lA, 2C and 4A). They are ubiquitous in all tree- leaf and forest soil isolates examined and comprise 5-- 10 per cent of the total opal. Spheres range continuously in dia. from 1 to 50 μ m; the larger sizes correspond to $> 5 \mu m$ opaline isolates. Most lack surface detail even at magnifications up to $30,000 \times$, but some have slight indentations or protrusions which likely mark sites of former host attachment (Figs. IB and C and 2C). In many cases they tend to assume nearly perfect spherical form. Based on recent work by Geis (1973) the spheres are probably formed as vesicular infillings in cell lumen or vesicles budding from a silicifying cell wall. Similar interstitial voids have been reported in birch *(Betula)* woody tissues (Drum, 1968). Spheres are relatively stable, even in \lt 5 μ m sizes; they occur in $2-5 \mu m$ soil opaline isolates (Fig. 1C), are observed in random scans of total soil clay fractions (Fig. ID), and are conspicuous in residues of tree-leaf isolates subjected to alkali dissolution. They resemble sphere-like aggregates of Anna kaolinite as reported by Bohor and Hughes (1971), but the latter have much coarser-textured surfaces than opaline spheres. Such similarities do emphasize the need to couple SEM

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Fig. 1. Opaline spheres observed in 2-5 μ m isolates of American beech leaves (A and B); 2-5 μ m opaline isolates from a forest soil (C) and the total clay fraction ($<$ 2 μ m) of a forest soil (D).

observations with other mineralogical tools for positive specimen identification.

Cup structures are another easily identified form in the 2-5 μ m opaline fraction of most vegetative (Figs. 2A-C) and soil (Fig. 2D) isolates. Less frequently cups occur in the $\langle 2 \mu m \rangle$ leaf isolates but none were observed in a soil clay fraction. Because many of these forms persist after alkali dissolution, their absence in clay fractions is attributed more to a lack of synthesis in clay-size units than to instabtlity. Most individual cups range in dia. from 2 to 10 μ m. This range conforms to the geometry of silicified epidermal or mesophyll cell elements. Walls are generally only about O· J- 0.2μ m thick and range in surface texture from fine

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(Fig. 2A, B and D) to coarse (note wall of cup fragment in Fig. lA and cup assemblage, Fig. 2C). On some of the latter a veined pattern occurs (Fig. 2C) and often the aggregate motif of the structure is revealed (Fig. lA). Shield-shaped cupped fragments (Fig. 2D, lower center) are common components of American beech isolates and may represent lower surfaces of inverted cups similar to Fig. 2A. Frequently, cups are partially filled with opaline detrital fragments (Fig. 2D) or *in situ* silica infillings of mosaic tabular aggregates.

Scrolled, convoluted, and twisted sheets of opal comprise 2/3 to 3/4 of the opal observed in \lt 5 μ m leaf. isolates of all species (Fig. 3). They range widely in geometry, but some resemble horns (Fig. 3A) and scrolled

Fig. 2. Opaline cup structures observed in 2-5 μ m leaf isolates of American beech (A); sugar maple (B); American linden (C) and 2-5 μ m opaline isolates of a forest soil (D). Note sphere (s) in (C).

sheets (Fig. 3B) while others are nondescript fragments (Figs. 3C and D). Characteristically, the sheets are thin $($0.1 \mu m$), coarse-textured (indicative of their compo$ site aggregate structure), and often exhibit veined surface patterns (Fig. 4B). In spite of the fact that some of these fragile forms resist alkali dissolution, they comprise a disproportionately small percentage of the 2- $5 \mu m$ soil opal isolate. Either these structures are fragmented beyond recognition after deposition in the soil or they are readily solubilized; the latter seems more plausible considering these forms have a relatively large specific surface subject to dissolution.

Rods represent another important component of $<$ 5 μ m opal fractions. They range from irregular

trough (Fig. 4A), sheath (Figs. 4B and 6B, lower center) and helical spiral (Fig. 4C) structures to more solid bodies with ribbed axial (Fig. 4D) or paired lateral protrusions (Fig. 4E); the latter probably represent sites of former sphere attachment. Others lack characteristic form or markings. They likely represent silicified bundle sheath parenchyma cells (Figs. 4B and 6B) and vascular elements (Fig. 4C, xylem). Rods commonly range from $1-2 \mu m$ in width to $8-10 \mu m$ in length. Many are optically anisotropic (Fig. lOA) with parallel or, less frequently, aggregate extinction. These structures appear to be relatively stable as evidenced from their ubiquitous occurrence in $2-5 \mu m$ soil opal isolates and alkali dissolution residues (Figs. lOA and B).

Fig. 3. Scrolled and convoluted sheets of opal observed in 2–5 μ m leaf isolates of American beech (A); sugar maple (B) ; white ash (C) and slippery elm (D) . Note associated sphere (s) and veined surface patterns (v).

Morphologically it will be difficult to differentiate tabular mosaic forms of opal (Figs. 4D and 5) from aggregates of phyllosilicate clays (Fig. 1D). White ash, American beech, slippery elm and sugar maple leaves contribute tabular bodies of opal similar to those forms observed along with the ribbed rod illustrated in Fig. 4D. These structures are very similar to such bodies isolated from the opal fraction in soils (Fig. 5). Some of those isolated from the soil show considerable evidence of point bridging (Fig. 58. b) with adjacent opal bodies and solution pitting and etching (Fig. 58, pl. Perhaps some of the mosaic aggregates found in soils which consist of a loose-packed, low order arrangement of tabular platelets are a consequence of

post depositional cementation of tabular opal fragments. This phenomenon may also explain assemblages of unrelated opaline forms sometimes observed in soil opaline isolates. However, this does not preclude the probability that many of the mosaic aggregates are from silica infillings in epidermal cells.

Silicified guard cells of stomata (Fig. 6) are a very distinctive form in most vegetative isolates. Similar forms were seldom observed in soil isolates and thus their stability under these forest weathering conditions is in question.

Crystalline silica phases co-associated with opal

At the inception of this study. opal of forest origin

Fig. 4. Opaline rod-like structures observed in 2-5 μ m leaf isolates of American linden (A), sugar maple (B) and American beech (C); $< 2 \mu m$ leaf isolates of sugar maple (D) and $2-5 \mu m$ opaline isolates of a forest soil (E). Note the tabular bodies in (D) and trough-shaped rod in (A) with attached spheres (s).

was considered more susceptible to crystalline conversion upon aging in the soil than grass opal (Wilding and Drees, 1972). Evidence for this hypothesis consisted of the numerous optically anistropic domains intimately associated with isotropic phases in opal extracted from forest soils. The optical effects were similar to those in Figs. 10A and 11B and were assumed to represent crystalline silica pseudomorphs after opal. An alternate explanation for these observations is cosynthesis of crystalline silica phases with opal in leaf tissues prior to their incorporation in the soil. The following discussion will consider evidence for the latter hypothesis.

X-ray diffractograms of the $<$ 2 μ m and 2–5 μ m silicate isolates derived from various tree leaves are presented in Figs. 7 and 8. All patterns illustrate a broad reflection in the region of *22° 20.* This band is associated with submicroscopic crystallites of cristobalite in low order arrangement. Species vary in their content of crystalline phases, namely cristobalite at 4·05 A and α quartz (chalcedony) at 3.34 and 4.26Å. American linden, slippery elm, and white ash contain more quartz and cristobalite in both $2-5 \mu m$ and $\lt 2 \mu m$ isolates. It is also evident that greater amounts of these crystalline phases occur in the $2-5 \mu m$ fraction (Fig. 8) than in the $\langle 2 \mu m \rangle$ separate (Fig. 7). This corresponds

Fig. 5. Tabular forms of opal isolated from the $2-5 \mu m$ fraction of a forest soil. Note the sphere (s) in (A) and point bridges (b) and solution pitting and etching (p) in (B).

to the large number of birefringent $2-5 \mu m$ rods and equidimensional bodies found in alkali dissolution residues of tree leaves (Fig. lOA). Refractive indices of most birefringent rods range from 1·53 to 1·55 indicative of quartz. Powder camera diffractometry of these residues confirm α quartz as the major crystalline component with secondary or trace quantities of cristobalite.

X-ray diffractograms are presented for $2-5 \mu m$ soil

opaline isolates in Fig. 9. Distinct peaks are noted for both cristobalite and quartz which compare favorably with corresponding peaks in vegetative isolates. Optical observations indicate that most of the crystalline forms are of characteristic biogenic origin.

No differences were noted in either optical or X-ray diffraction analyses of tree-leaf isolates prepared by the two alternate methods. Neither the form nor abundance of co-associated crystalline phases were altered

Fig. 6. Silicified stomata guard cells isolated from sugar maple (A) and American beech (B). Note sheathlike rod in lower center of field.

White Oak 26 **DEGREES 28** Fig. 7. X-ray diffractograms of $\lt 2 \mu m$ silicate isolates de-

Hackberry __ ..JJiI\'OIofMlffl~~ *....*

Fig. 8. X-ray diffractograms of $2-5 \mu m$ opaline (< 2.3 sp. g) isolates derived from various tree leaves. Designations QTZ represent quartz and CR cristobalite.

Fig. 9. X-ray diffractograms of the $2-5 \mu m$ opaline isolate obtained from the surface (AI) horizon of forested Miamian soils at several different locations. Designations QTZ represent quartz and CR cristobalite.

by ashing at temperatures of 300-400°C as contrasted to low-temperature ashing. Thus, it is concluded that quartz and cristobalite are synthesized in tree leaves as a natural metabolic function and do not represent artifacts produced by isolation procedures. Apparently, silicon is taken up by the tree in the form of monomeric silicic acid along with other nutrients from soil solution, transported via vascular elements to epidermal leaf tissues, and finally deposited in preferred loci in the form of amorphous and crystalline silica phases, all within a time span of less than a year. The precise physiological mechanism or energy transfer involved is unknown. Likewise, to what extent inorganic and organic components prevalent in live tissues foster the rate of nucleation or nature of silica polymorphs formed is in question.

Alkali dissolution of tree-leaf isolates

Between 2/3 to 3/4 of the total opal isolated from tree leaves can be dissolved in a 2'5-min digestion in boiling 0·5 N NaOH (Table 1). Jones (1969) found that only about $1/3$ of a $20-50 \mu m$ grass opal specimen could be dissolved in a 20-min digestion in boiling 0·5 N NaOH; the reaction followed first order kinetics. By calculation, only 5 per cent of the grass opal would be dissolved in a 2·5 min digestion period. This suggests

TREE - LEAF

Amer. Linden

Slippery Elm-

Amer. Beach

Mopk

White Ash

Sugar

ISOLATES «2/1om) QTZ QTZ

3.34 م $405A$ 1

CR

Species	Size fraction (μm)	Weight lost $\binom{9}{0}$
American beech	$5 - 20$	92
	Total	78
White oak	> 50	85
	$5 - 20$	85
	Total	60
Sugar maple	> 50	54
	$5 - 20$	64
	Total	63

Table I. Alkali dissolution of opaline constituents isolated from various deciduous tree leaves

that the rate of dissolution for forest opal is considerably greater than for grass opal. Greater rates of dissolution for forest opal by a factor of 10-15 probably reflect morphological differences between forest and grass opal. Forest opal consists of thin sheet-like incrustations of cellular components with high specific surface whereas grass opal consists mainly of solid polyhedral structures that represent more complete infillings of epidermal cells. Susceptibility to dissolution of forest opal is a function of species and particle-size, with American beech, white oak and sugar maple least resistant in that order (Table I). Smaller proportions of the opal in the total fraction from American beech and white oak dissolved than in the $5-20 \mu m$ isolates. This difference is attributed to more resistant $\lt 5 \mu m$ components. These two species contain only 5-10 per cent of their total opal in $> 20 \mu m$ separates (Wilding and Drees, 1971); therefore, lower total dissolution

cannot be attributed to this fraction. More resistant $<$ 5 μ m components are also consistent with optical observations of residues which contain many anisotropic and isotropic bodies in the $<$ 5 μ m range (Fig. 10A). Lower dissolution rates for white oak and sugar maple may reflect their higher organic carbon status as inferred from their dark brown to black color. To what extent occluded carbon or possibly chemically bonded forms.protect opal against such attack is unknown, but the possibility exists that such impurities may serve to stabilize the opal and retard its rate of dissolution.

Many of the residues range continuously from being optically isotropic to anisotropic. This suggests they vary continuously from short range order (optically isotropic and X-ray amorphous) to long range order (optically anisotropic and X-ray crystalline) as previously proposed for opal of geologic origin by Jones *et al.* (1963).

Optical and SEM photomicrographs of selected (outlined) $> 5 \mu m$ grains of sugar maple (Fig. 10) and white oak (Fig. 11) indicate that the crystalline phases (birefringent zones, Figs. lOA and II B) are not clearly associated with any particular discrete morphological element of the structure. In both cases the crystalline phases appear to be thin, coarse-textured tabular or slightly scrolled silica sheets. The small rod-shaped crystallites and sphere (Fig. II C, rand *s* respectively) do not appear birefringent, at least in the geometric orientation photographed. In spite of the dissolution treatment these residues do not illustrate much evidence of solution pitting or etching.

Contributions of forest opal and co-associated crys-

Fig. 10. Alkali dissolution residues of sugar maple as observed under cross polarized light (A) and under the SEM (B) of the grain outlined in (A).

Fig. II. Alkali dissolution residues of white oak as observed under plane polarized light (A), cross polarized light (B) and under the SEM (C) of the grain outlined in (A) and (B). Note sphere (s) and rod-shape crystallites (r) associated with silicate sheets.

talline phases to clay fractions of soils have profound implications on the accuracy and interpretations of clay mineral analysis by wet chemistry techniques (Alexiades and Jackson, 1966). For example, opal would confound interpretations of amorphous components or kaolinite and halloysite determined by NaOH dissolution and based on dissolved $SiO₂$ or $SiO₂/Al₂O₃$ ratios. This problem would be more serious for surficial horizons of forest zones which had not been subjected to intense chemical weathering. Under these conditions substantial amounts of opal would be expected to accumulate in the clay-size fraction. Much of the opal would be dissolved in a 2'5-min digestion of boiling 0·5 N NaOH.

Biogenically derived quartz could also confound the use of this mineral as a stable reference index for gainslosses reconstruction studies. In this conjunction the question is raised whether biogenic quartz may explain the concentration of clay-size quartz in surficial zones of most forest soils; this anomaly has gener- . ally been attributed to contamination of the surface from aeolian deposits.

CONCLUSIONS

The following conclusions are drawn from this preliminary work:

I. Opal derived from deciduous tree leaves can be identified by SEM in \lt 5 μ m fractions of soil opaline isolates. Spheres, cups, and scrolled or convoluted sheet forms are most distinctive.

2. Crystalline silica in the form of α quartz and cristobalite is co-associated with amorphous opaline phases in $\lt 5 \mu m$ tree-leaf and soil isolates. These crystalline components are apparently synthesized within vegetative tissues as a function of life metabolism and subsequently deposited in the soil along with the amorphous opaline phase.

3. Approximately $2/3-3/4$ of tree-leaf opaline isolates are dissolved in a 2'5-min digestion of boiling 0·5 N NaOH. Rate of dissolution for forest opal is lO-IS times greater than grass opal. This difference probably reflects differences in morphology; the forest opal consists of incrustations of cellular components with numerous thin sheet structures with high specific surface while grass opal consists mostly of solid polyhedral structures from opaline infillings of epidermal cells.

4. Alkali dissolution residues represent a continuum from optically isotropic to optically anisotropic bodies. This continuity suggests that vegetative opaline constituents range continuously from short range (amorphous optically and by X-ray diffraction) to long range order (optically anisotropic with sharp X-ray diffraction peaks of quartz and cristobalite.)

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Résumé—Le microscope électronique à balayage (SEM) est très utile pour l'identification de l'opale biogène. Des sphères d'opaline, des cupules et des feuillets en volutes ou enroulés, ont été identifiés à la fois dans des fractions isolées à partir de sols ou de végétaux. L'analyse par diffraction X indique que du quartz alpha et de la cristobalite sont associes avec la phase opale amorphe synthetisee durant Ie metabolisme des feuilles d'arbres a feuilles caduques. De telles phases cristallines sont les plus abondantes dans la fraction 2-5 μ m, et beaucoup d'entre elles consistent en des bâtonnets anisotropes à extinction parallele ou en des masses equidimensionnelles avec une extinction d'aggregat. De 2/3 a 3/4 de I'opale totale isolée des feuilles d'arbres à feuilles caduques est solubilisée lors d'un traitement de 2,5 min dans NaOH 0,5 N bouillant. La vitesse de dissolution est [onction de la taille des particules et de I'espece de l'arbre. L'opale biogene forestiere est environ 10-15 fois plus soluble que l'opale venant de l'herbe. ce qui indique probablement une surface spécifique plus élevée pour la première.

Kurzreferat-Das Rasterelektronenmikroskop (SEM) ist brauchbar für die Identifizierung von biogenem Opal. Kugeln, Schalen und gerollte oder gewundene Blattchen aus Opal wurden sowohl in Fraktionen aus Boden als auch aus Pflanzematerial nachgewiesen. Die Rontgenbeugungsanalyse zeight, daB sowohl α -Quarz als auch Cristobalit mit der amorphen Opalphase vergesellschaftet sind, die im Verlauf des Lebensstoffwechsels von Laubbaumblattern synthetisiert wird. Soiche kristallinen Phasen traten am häufigsten in der 2-5 μ m-Fraktion auf und bestehen vielfach aus anisotropen Stäbchen mit paralleler Auslöschung oder aus Körpern mit gleichen Achsenlängen und Aggregatauslöschung. Zwischen 2/3 und 3/4 der gesamten aus Laubbaumblattern isolierten Opalfraktionen losen sich nach 2,5 miniitiger Behandlung in kochender 0,5 N NaOH. Die Auflösungsrate war eine Funktion der Korngröße und der Baumart. Biogener Opal forstlichen Ursprungs war etwa 10-15 mal löslicher als Grass-Opal, was wahrscheinlich die höhere spezifische Oberfläche des erstgenannten widerspiegelt.

Резюме — Растровый электронный микроскоп полезен при выяснении природы биогенного опала. Опаловые шарики, чашечки и витковые или свернутые листки найдены как в земляных, так и в растительных выделениях. Рентгенографический анализ показывает, что как альфа квартц, так и кристобалит связаны с аморфной опаловой фазой, образовавшейся во время жизненного метаболизма листьев лиственных деревьев. Эти кристаллические фазы обильнее всего встречаются во фракции 2-5 μ м и могут состоять из анизотропических палочек с параллельной экстинкцией или с телами одинаковой размерности с экстинкцией агрегата. Между 2/3 и 3/4 общего опалового выделения из листьев лиственных пород растворилось при вываривании в течение 2,5 минут в кипящем 0,5 N NaOH. Скорость растворения зависит от размера частины и от нороды дерева. Биогенный опад лесного происхождения растворяется в 10-15 раз скорее, чем травяной опал, что, возможно, отражает более высокую удельную поверхность nepBOro.