SEQUENTIAL ACTIVE ALTERATION OF RHYOLITIC VOLCANIC ROCK TO ENDELLITE AND A PRECURSOR PHASE OF IT AT A SPRING IN MICHOACAN, MEXICO

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Abstract – Sequential alteration of volcanic rock of rhyolitic composition to a precursor of endellite, and thence to tubular endellite, may be observed at a hot spring about 27 km north of Ciudad Hidalgo, Michoacan, Mexico. Ojo Caliente de Laguna Larga (or Verde) yields H_2S -SO₂ containing water, temperature about 45°C, pH 3·5–3·7, from multiple orifices in vesicular and slabby rock with which the water reacts.

First-stage alteration of the rock consisted of permeation and dissolution, accompanied by deposition of clay substance in vesicle walls. The clay substance, although "amorphous" to X-rays, yields a weak DTA of endellite, and kaolinite. It is interpreted as a precursor of endellite. Further reaction included continued filling of vesicles and spreading argillation which coalesced between vesicles and other rock pores. Ultimately, the rock was completely "digested" to well-defined endellites.

Strong desilication and depotassication of the rock is indicated by the dissolved substances in the spring water: Si 59·1 (equivalent to 127 SiO₂) ug/ml: Al, 1·63; Fe, 0·65: Mg, 0·21: Ca, 178: Na, 0·73: K, 13·7. Despite high K⁺, predominantly high H⁺ and Al³⁺ induce formation of endellite. The claywater system accords well with stability diagrams.

Allophane-endellite in Indiana and endellite at Stanford, Kentucky, are formed from cold, strongly acid, meteoric-water solutions high in Si and Al relative to kaolinite dissolved in water. Factors of genesis common to both hot- and cold-water endellite are cited, suggesting that they are premissively critical, but not necessarily restrictive, to the origin of endellite.

INTRODUCTION

ACTIVE alteration of volcanic rock to tubular endellite and a somewhat indefinite precursor of endellite has been traced sequentially at a hot spring, Ojo Caliente de Laguna Larga (or Verde). The spring, flowing sulphurous water at 45°C, is located on the periphery of the Los Azufres thermal area, about 240 km west of Mexico City, and about 27 km north of Ciudad Hidalgo, Michoacan, Mexico. The clay deposit described by Thomas L. Kesler at the 18th Clay Minerals Conference occurs in this area. The composition of the spring water, which is high in dissolved Si, Al, and K, is plotted in stability diagrams within the kaolin area, but close to the boundary with amorphous silica. Such composition accords well with geologic observations elsewhere in Mexico of ancient hydrothermally formed kaolin deposits commonly capped with silica gossans.

The endellite-producing geochemical environ-

one which produces endellite at earth-surface temperatures from action of acid solutions on a kaolinitic residuum from limestone. Properties, such as high H^+ concentration (even though K^+ is high) and high Al^{3+} , which are in common between the two occurrences, may be essential in the genesis of endellite.

ment at this hot spring holds certain similarities to

THE SOLID COMPONENTS OF THE ALTERATION SYSTEM

An essentially continuous series of specimens collected from almost-fresh rock to massive clay permits study in detail of the changes in the solid phases during alteration. The original country rock is rhyolitic in composition, analysis (a), Table 1. It is predominantly glassy, with sporadic phenocrysts of quartz and glassy feldspar. Many spherulites, 0.5-1.0 mm in diameter, developed in it are now devitrified into radially-oriented or plumose, microfibrous minerals. Such fibers may also be radially oriented in the walls of vesicles, commonly 3–5 m in diameter, which comprise about 25 per cent of the rock. In some rock layers the vesicles are aligned with the flow structure, but where maximum

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	a*	b†	c‡
SiO ₂	71.95	65.41	44.64
Al_2O_3	18.18	21.37	34.89
Fe_2O_2	0.86	n.d.	2.00
TiO ₂	0.06	n.d.	0.10
MgŌ	0.65	n.d.	0.01
CaO	0.96	n.d.	0.09
Na_2O	1.26	n.d.	0.32
K ₂ Ō	4.02	n.d.	0.06
SŌ₄	n.d.	n.d.	0.82
P ₂ O ₅	0.06	n.d.	0.08
H ₂ O ⁻	0.25	0.52	2.60
$H_2O^+(105^\circ)$	1.70	9.70	14.30
	99.95	97.00 (partial)	99-91

Table 1. Analyses of parent rock and clay at Ojo Caliente de Laguna Larga of the Los Azufres Thermal Area, Michoacan, Mexico

*Relatively fresh volcanic parent rock. †Clay filling vesicles.

‡Massive clay, final alteration product.

Analyses by Bruce Williams Laboratory.

argillation is observed, they are scattered randomly.

The walls of the vesicles typically are lined with a thin, white, sugary, microcrystalline coating of low-cristobalite. On the walls of many vesicles are large blebs of low-cristobalite, 0.2-1 mm in diameter, whose surface structure resembles "microcauliflower".

Earliest alteration is manifested by the development of thin rinds of clay substance underlying the cristobalite linings of the vesicles, and dulling of luster throughout the rock. Progressively more alteration is shown by specimens in which the clay in the vesicle walls is much thicker-the thickening being due to both clay-filling inward, and clay alteration that has moved outward into the enclosing rock. Part of the filling inward is apparently due to volume increase as "rock" is transformed to less-compacted clay; evidence for this interpretation is the remnant cristobalite blebs perched on the inward-migrating clay walls. Other portions of the inward-filling appears to be due to later deposition of clay, presumably as a later precipitate or crystallization from solution. Evidence for the late filling is that the clay substance overlies and surrounds the cristobalite, known to be early. Eventually clay fills completely and packs tightly in the filled vesicles. The filled vesicles are more common in rock that is threefourths, or more, altered.

The clay substance filling the vesicles presents a problem in identification. It is essentially amorphous

to X-rays, yielding no significant reflections above background, including the region of intense reflections by basal spacings. From this criterion it might be called allophane.

Differential thermal analysis of it, however, yields weak to moderate peaks (endothermic maximum at 515°, exothermic at 980°) characteristic of endellite (crystalline). Its i.r. spectrogram is of the kaolinite type, but it lacks one prominent kaolinite absorption at about 528 cm⁻¹, an Si-O bonding, Fig. 1. Both the vesicle-clay, and the well-defined endellite that is the end-product of alteration, lack, or show scarcely preceptible development of, the two OH-absorptions at about 3655 and 3670 cm⁻¹ in kaolinite.



Fig. 1. Direct photo-copy of i.r. spectrograms, across 4000-3400 and 700-250 cm⁻¹ wave numbers, of vesicle clay and endellite, Ojo Caliente de Laguna Larga, and Keokuk kaolinite. Note increased absorptions by better crystallized clay minerals at 3600-3700, and 500-550 cm⁻¹ wave numbers.



Fig. 2. Transmission electron micrographs of endellite from Ojo Caliente de Laguna Larga. (a) Clay filling from vesicles in rock. (b) Massive clay, final argillation.

Under the petrographic microscope a sedimented sample of the vesicle-clay disperses in refractive index oils as flocs or aggregates having fibrous structure inherited from the fibrous spherulites. Judging from index of refraction, more than one substance is present in the fibers and flocs, but individual fibers are too tiny and intimately mixed to be measured for refractive index. In a 1.510 oil much of the fibrous-material appears to be about 1.480 or 1.490 in refractive index. In a 1.485 oil, much of the material appears to be about 1.510-1.520 in index. The material is apparently isotropic, except that thick clots viewed between crossed polars and a first-order red plate may show slight non-red color, but without distinguishable orientation. From refractive indicies, it could be fibers of allophane hydrated to different degrees, or a clay substance mixed with micro-fibers of cristobalite, or a transition phase, i.e. precursor, to endellite.

Transmission electron micrographs of the watersuspended clay show well-developed endellite morphology that is similar to the fully altered clay, Fig. 2(a). This is anomalous with the other noncrystalline properties. Since granular allophane was not observed with the electron microscope it may be absent, or alternatively, it may have suspended so poorly in relation to endellite that it escaped recovery on the EM grids.

A partial chemical analysis of the vesicle clay is given as (b) in Table 1. Al and ignitionwater are not in formula-ratio for either endellite or halloysite. Silica is stoichiometrically higher than alumina for any kaolin mineral, but some free silica in excess of the kaolin formula is known to be present as cristobalite. Chemically, the vesicle clay could be allophane of high-SiO₂: Al_2O_3 ratio, or allophane plus cristobalite (or possibly chalcedonic silica).

Summarizing from X-ray and optical criteria the clay material appears to be "amorphous". From DTA and i.r. it exhibits part of the properties of endellite. Alternative classification of it might be allophane, a mixture of allophane and endellite, or a precursor of endellite. The latter seems most reasonable now.

Endellite, the final clay-alteration product. Complete alteration of the rock yields massive, grayish, plastic clay entirely devoid of rock or mineral relict structure or materials. The X-ray diffractogram records only endellite that is partially dehydrated and collapsed, Fig. 3.

Transmission electron micrograph of it show typical endellite tubules or scrolls, Fig. 2 (b). Its chemical composition is given in analysis (c), Table 1. This composition accords well with that expected of a room-dry endellite plus sulphate – which is consistent with the "sulphur water"



Fig. 3. X-ray diffractogram of endellite from Ojo Caliente de Laguna Larga. Basal spacing is undergoing collapse from 10A to 7A. Ni-filtered, Cu K_{α} radiation.

present in the plastic, wet clay mass as collected. If the weights of iron, sulphate, and low-temperature moisture are redistributed to alumina and silica, the analysis would approach the ideal formula for halloysite. Alkali and alkaline earth metals present in the parent rock have been typically leached away.

After tracing the evolution of original and secondary structures of the rock and minerals during argillation, the massive, uniform, earthy texture of the final clay is impressive. Not only are no relic structures preserved but no residual quartz, cristobalite, or other duaghter minerals remain. A complete "digestion" of the rock to clay has taken place. Such complete changes have been previously documented from the flint clay at Estola (Hanson and Keller, in press, this journal), and noted, but without special mention, at most of the refractory clay deposits in Mexico. Transformation "digestion" of a characteristically structured parent rock to massive clay devoid of parent structure is one of the profound processes in geology.

THE SPRING-WATER SOLUTION

The water from the spring, Ojo Caliente de Laguna Larga was measured at 45° C (uncalibrated thermometer), pH 3.5 with Hydrion paper in the field on July 1, 1970, and pH 3.7 with a glass electrode in the laboratory several days later. An odor of H₂S-SO₂ rose from the water, and white clay coated the rocks damming up a pool at the spring site.

A sample of the water collected and shipped in a polyethylene bottle to the laboratory was analyzed four days later yielding analysis (d), Table 2. Strong desilication and depotassication of the rock by dis-

Table 2. Analyses of solutions associated with endellite and kaolinite, expressed in micrograms per milliliter, essentially ppm

	d*	e†	f‡	g§
Si	59·1	3.46	16-2	96.3
SiO ₂ (equiv.)	127	7.4	34.7	206
Al	1.63	0.01	30	1.53
Fe	0.65			1.95
Mg	0.21	0.09		1.35
Ca	0.78	0.95		4.68
Na	0.73	0.26	6.4	65.8
K	13.7	0.30	1.28	22.8
SO₄			150	
pH	3.5-3.7	5.85	3.6	3.9
Temp.	45°C	$25^\circ \pm C$	$25^{\circ} \pm C$	74°C

*Spring water, Ojo Caliente de Laguna Larga, Los Azufres Thermal Area, Michoacan, Mexico.

†Solution, essentially stabilized, of kaolinite, Murfreesboro, Ark., in distilled water.

‡Solution extracted from plastic endellite, Stanford, Ky., in distilled water.

§Spring water from a major spring in the Central Los Azufres Thermal Area, Michoacan, Mexico.

solution obviously took place. This leachate from the rock would have been an alkaline, potassium silicate solution if the sulphur compounds had not been present to acidify it. No doubt the acidity, pH 3.5-3.7, was essential in dissolving the high concontration of Al (certainly high in comparison to that typically dissolved from kaolinite in distilled water at room temperature) analysis (e), Table 2. At pH 3.5, ions of Al are predominantly Al³⁺ (Reesman, Pickett, and Keller, 1969).

The high concentration of K^+ in an Al–Si solution system, such as this one, might be expected to induce the formation of mica, but the concentration of H^+ is also high enough to lead to endellite. The composition of this spring water, where located by the square symbol on stability diagrams of clay and associated minerals, occurs in the kaolinite field or on the boundary with amorphous silica, temperature considered, Figs. 4–7. The triangular symbol refers to another hot spring, and the circle to a solution from Kentucky endellite, both discussed in foregoing paragraphs.

The deposition of clay from solution has been emphasized more in this report than that of silica. Nonetheless, at many other large refractory-kaolin deposits in Mexico, where hot-water activity has long since ceased, voluminous quartz and chalcedonic-silica gossans cap the clay bodies. Slight shift in temperature, or composition, or quartzseeding of silicate loaded solutions rising from kaolin beneath to the colder earth's surface, would result in deposition of silica gossan. For a geo-



Fig. 4. Stability diagram relating pH and H_4SiO_4 at 25°C and 95°C. Composition of spring water from Ojo Caliente de Laguna Larga, shown in the square, is on the boundary between kaolinite and silica at 25°C but within kaolinite at a higher temperature. Los Azufres water, triangle, lies closer to the silica field. Kentucky-endellite solution, circle, is in the alunite field. Diagram modified after Raymahashay, 1968.

chemical example, at another hot spring, 74° C, in the nearby central Los Azufres area, where thermal activity was more intense than that at Laguna Larga, *no* clay was observed. An analysis of the spring water (g), Table 2, plotted on the stability diagrams by triangles showed that silica – not kaolin – would most likely be the solid phase stable in the spring water. Rock samples collected at the site are partly silicified. Can this be a gossan in process of formation and clay being formed below?

If sulphate had been more concentrated at either of these springs, alunite might have been deposited, Fig. 4, as it does occur in many other hot-spring kaolin deposits.

ENDELLITE DEPOSITED FROM SOLUTION

As previously discussed, some endellite was deposited from solution in the vesicles of the rock at this hot-spring locality. It was produced in large quantity during the final digestion of the rock. These depositions occurred at elevated temperatures.

Endellite has been deposited from solution also at low, i.e. earth-surface, temperatures. At Gardiner



Fig. 5. Stability diagram relating K^+/H^+ , H_2SiO_4 at 25°C. Composition of spring water from Ojo Caliente de Laguna Larga, symbol in square, is in the "Amorphous Silica" field just outside "Kaolinite", which presumably would be off-set to the right for 45°C. The Los Azufres spring water at 74°C, shown in the triangle is farther away from kaolinite, in the silica field. The Kentucky endellite solution, in circle, is clearly in the kaolinite field. Diagram modified after A. B. Carpenter (personal communication).

Ridge Mine, Indiana, allophane and endellite were deposited from cold, acid sulphate solutions percolating downward through the Spice Valley Member of the Mansfield Sandstone (Callaghan, 1948; Sunderman, 1963). Near Stanford, Kentucky, endellite was (and is) added to "growing" concretions today by cold surface-water solutions (Keller, McGrain, Reesman and Saum, 1966).

At the Stanford locality, rain water descending through the New Albany Shale oxidized iron sulphide, producing acid solution at pH 1 to 2 (field measurement). Such acid waters also dissolve Si and Al from illite in the shale and continue downward into an ancient, kaolin-rich residuum from the weathered Brassfield Limestone. Further "digestion" of the residuum, plus precipitation and/or replacement in it, is culminating in concretions of endellite "growing" in it today. An analysis of the solution from this plastic endellite suspended in



Fig. 6. Stability diagram relating Na^+/H^+ , H_4SiO_4 at 25°C. The Mexican hot-spring waters, square and triangular symbols, are on or near the boundary between kaolinite and amorphous silica areas. Kentucky-endellite solution is well within the kaolinite area. Alunite is commonly accessory in many of the Mexican hydro-thermal kaolin deposits. Diagram modified after Feth, Roberson, and Polzer (1964).

distilled water is given in (f), Table 2.

Comparisons of the hot- and cold-water solutions from which endellite has crystallized show the following conditions to be held in common.

1. Endellite has been precipitated out of a solution phase. Keller and Hanson (1969) speculated that endellite had crystallized at Sombrerete, Zacatecas, Mexico from cooling solution, rather than from *in-situ* conversion of a solid.

2. The pH of both solutions was in the range $3 \cdot 5 - 3 \cdot 7$.

3. Because of the low pH, the ions of Al in both solutions were predominantly Al^{3+} (Reesman, Pickett, and Keller, 1969).

4. The concentrations of Si and Al in solution were very high relative to those in a solution of kaolinite in water.

5. The solvent anions were sulfate, plus lessoxidized sulphur compounds.

6. These properties prevailed although the



Fig. 7. Stability daigram relating pH-1/3 Al³⁺ and pH_4SiO_4 at 25°C. Compositions of both Mexican hotspring waters lie in the field not exclusively assignable to either kaolinite or halloysite. The Kentucky endellite solution, in circle, is farther in the silica area than would be expected from its field occurrence. Diagram from Kittrick, J. A. given at Seminar lectures, University of Missouri, Columbia (1970).

parent rocks were radically different, i.e. volcanic rock vs. sedimentary (shale and an ancient residuum).

The geochemical conditions listed above are thought to be soundly permissive for the origin of endellite. To offer them as being restrictive of conditions possible for endellite genesis when based on only two occurrences, would be geologically presumptuous, in our opinion. To understand comprehensively the geochemical geological environments which determine whether allophane, endellite, halloysite, disordered kaolinite, wellcrystallized kaolinite, or dickite originate, and are stable (or reversible) may require many observations. We offer these two in progress.

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Résumé – L'altération séquentielle d'une roche volcanique de composition rhyolitique en un précurseur de l'endellite puis en une endellite tubulaire, peut-être observée dans une source thermale, à environ 27 km au nord de Ciudad Hidalgo, Michoacan, Mexique. Ojo Caliente de Laguna Larga (ou Verde) fournit une eau contenant H_2S et SO₂, à une température d'environ 45°C et à pH 3,5-3,7; cette eau s'échappe des multiples orifices d'une roche vésiculaire et stratifiée; elle réagit avec la roche.

La première étape de l'altération de la roche consiste en une pénétration et une dissolution, accompagnées du dépôt d'une substance argileuse sur les parois des vésicules. La substance argileuse, quoique "amorphe" aux rayons X, donne, en ATD, une courbe peu marquée d'endellite et de la kaolinite. Ceci est interprété comme un précurseur de l'endellite. Une réaction plus poussée entraîne le remplissage complet des vésicules et l'extension de l'argilification, qui soude les vésicules aux autres pores de la roche. A la fin, la roche est totalement "digérée" en endellite bien définie.

L'appauvrissement intensif de la roche en silice et en potassium ressort de l'analyse des substances que l'on trouve dissoutes dans l'eau de la source: Si 59,1 (équivalent à 127 SiO₂) μ g/ml: Al 1,63; Fe, 0,65: Mg, 0,21: Ca, 178: Na, 0,73: K, 13,7. En dépit de la forte teneur en K⁺, les teneurs élevées en H⁺ et Al³⁺ induisent d'une façon prédominante la formation de l'endellite. Le système argile—eau concorde bien avec les diagrammes de stabilité.

Le mélange allophane-endellite dans l'Indiana et l'endellite de Stanford, Kentucky, sont formés à partir de solutions d'eau atmosphérique froides et fortement acides, qui ont une forte teneur en Si et Al par rapport à la kaolinite dissoute dans l'eau. Les facteurs de la genèse communs aux endellites d'eau chaude et d'eau froide sont décrits, en suggérant qu'ils sont peut-être critiques, mais pas nécessairement restrictifs, pour l'origine de l'endellite.

Kurzreferat – Die aufeinanderfolgende Veränderung von vulkanischem Gestein Rhyolith-artiger Zusammensetzung zu einem Vorläufer des Endellith, und aus diesem zu rohrförmigem Endellith, kann an einer heissen Quelle etwa 27 km nördlich der Cidudad Hidalgo, Michoacan, Mexiko beobach-

tet werden. Ojo Caliente de Laguna Larga (oder Verde) liefert H_2S-SO_2 -haltiges Wasser, Temperatur ungefähr 45°C, pH 3,5-3,7 aus einer Anzahl von Öffnungen in blasigem und schiefrigem Gestein mit welchem das Wasser reagiert.

Die erste Veränderungsstufe des Gesteins bestand aus Durchdringung und Auflösung, verbunden mit Ablagerung tonigen Materials in den Bläschenwänden. Das tonige Material Erscheint wohl "amorph" under Röntgenbestrahlung, zeigt aber ein schwaches DTA von Endellith und Kaolinit. Es wird als ein Vorläufer des Endelliths angesehen. Die weitere Reaktion umfasste weitere Füllung der Bläschen und Ausbreitung der Tonbildung mit Kaoleszenz zwischen Bläschen und Gesteinsporen. Schliesslich war komplette "Digestion" zu wohlausgebildetem Endellith erfolgt.

Eine Analyse der in dem Quellwasser aufgelösten Substanzen deutet auf starke Entkieselung und Kaliumverlust des Gesteines hin: Si 59,1 (äquivalent 127 SiO₂) μ g/ml; Al 1,63; Fe 0,65; Mg 0,21; Ca 178; Na 0,73; K 13,7. Trotz hohem K⁺ Gehalt, wird die Bildung von Endellith in erster Linie durch hohe H⁺ und Al³⁺ Werte gefördert. Das Ton-Wasser System stimmt gut mit dem Stabilitätsdiagramm überein.

Allophan-Endellith in Indiana und Endellith in Stanford Kentucky werden aus kalten, stark sauren, meteorisch-wässrigen Lösungen, mit hohen Si und Al Gehalten im Verhältnis zu dem im Wasser gelösten Kaolinit, gebildet. Es werden Faktoren erwähnt, die der Entstehung von Endellith aus heissem sowie aus kaltem Wasser gemeinsam sind, und die als kritish für die Ermöglichung einer Endellithbildung, jedoch nicht unbedingt als beschränkend angesehen werden.

Резюме — Последовательное изменение вулканической породы риолитового состава до предшествующей энделлиту фазы и затем в трубчатый энделлит наблюдается вблизи горячего источника, расположенного около 27 км севернее Сьюдад Идальго (Мичоакан, Мексика). Источник Охо Кальенте де Лагуна Ларга (или Верде) дает содержащие H₂S-SO₂ воды с температурой около 45°C и pH 3,5-3,7, поступающие из сложного жерла в пористой и плитчатой породе, подвергающейся изменению.

Первая стадия изменения породы состоит в проникновении воды и растворении, сопровождающимся отложением глинистого вещества в пузырчатых стенках. Глинистое вещество хотя и является рентгено-аморфным, но дает слабую ДТА-картину энделлита и каолинита. Эта фаза интерпретирована как предшествующая энделлиту. Дальнейшее взаимодействие породы с водой включает постепенное заполнение мелких полостей и аргиллизацию породы между мелкими полостями и порами. В конце концов порода полностью «переваривается» в типичный энделлит.

Сильная десиликация и декализация породы устанавливается по составу воды источника (мг/мл): Si — 59,1 (эквивалентно 127 SiO₂); Al — 1,63; Fe — 0,65; Mg — 0,21; Ca — 0,178; Na — 0,73; K — 13,7. Несмотря на высокое содержание K⁺, преобладающе высокое содержание H⁺ и Al³⁺ приводит к образованию энделлита. Система глина — вода согласуется с диаграммами устойчивости. Аллофан-энделлит Индианы и энделлит Стэнфорда (Кентуки) образовались из холодных сильно кислых атмосферных вод с высоким содержанием Si и Al по отношению к каолиниту, растворенному в воде. Рассмотрены факторы образования энделлита из горячих и холодных вод, которые признаются возможными, но не единственными.