IMOGOLITE AND ALLOPHANE FORMED IN SAPROLITE OF BASALT ON MAUI, HAWAII*

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Abstract – Inorganic gel and allophane collected from basaltic saprolite on Maui, Hawaii, and studied by Patterson in 1964 were reexamined. The main constituent of the gel is imogolite, and gibbsite and allophane are the minor constituents. Electron and X-ray diffraction patterns, DTA curve, and an infrared spectrum of the gel are characteristic of imogolite. The allophane is virtually noncrystalline to X-rays but contains a small amount of imogolite in relatively short threads. High-resolution electron micrographs indicate differences in structural organization between allophane and imogolite and suggest crystallization of imogolite from allophane.

The occurrence of imogolite as a weathering product has been reported in many localities from pyroclastic materials but not from massive rocks. Probably the exceptionally high rainfall, excellent subsurface permeability of the weathered material, and the low pH and high organic content of the leaching solution provide favorable conditions for formation of imogolite from basalt on Maui.

INTRODUCTION

GELS and noncrystalline inorganic materials in soils and weathered rocks in Hawaii have been reported by several investigators (Tamura and others, 1953; Kanehiro and Whittig, 1961; Bates, 1962; Moberly, 1963; Lai and Swindale, 1969). The gel and associated allophane examined in the present study were collected and studied by S. H. Patterson (1964). The gel was approximately 90 per cent water. Both the dried gel and allophane are similar in composition, consisting of approximately 50 per cent alumina and 22–26 per cent silica, and most of the remainder was H_2O or other matter driven off when the material was heated at 1000°C. Patterson recognized that a small amount of gibbsite was present in the air-dried gel, but thought that both the gel and allophane are essentially noncrystalline to X-rays. He also interpreted the similarity in composition of the two materials and their marked differences from the associated saprolite as indicating related origin. The superposition and overlapping of the very hydrous gel on

allophane was thought to indicate the formation of allophane from the gel.

Later, an electron micrograph of the air-dried gel by E. J. Dwornik showed it to have a peculiar filiform morphology, and Patterson (1971) suggested that it is imogolite. The object of the present study is to verify this suggestion and to obtain further information about the relation between the gel and allophane. The exceptional purity of the material, which was suggested from the electron micrograph, and the fact that heretofore imogolite has been identified only in weathering products of pyroclastic materials prompt this report.

OCCURRENCE

The gel and associated allophane were collected from the face of a freshly bulldozed roadcut at a depth of 2–6 ft. The cut was on a trail under construction on the northwest slope of Haleakala volcano, East Maui. The materials occur as veins and as fillings and coatings on vesicles, vugs, and cracks in a dark-brown saprolite in the outer parts of joint blocks having cores of hard basalt. The allophane is white, soft, and open textured, and most of it when collected was covered by a clear or light-colored gel. Some of the gel is in clear irregular

masses ranging in size from microscopic specks to about one-fourth inch in dia. and is similar to a mass of frog eggs or thick gelatin. Some occurs as light-grayish-brown paper-thin laminae that extend along cracks for several inches.

METHODS OF INVESTIGATION

The gel had been preserved in water since its collection in 1960. The air-dried gel, which has the appearance of light-grayish-brown paper-thin laminae, and allophane were also examined. The undried gel was dispersed in water by treatment with the ultrasonic wave of 400 KC. Appropriate aliquots of the resulting viscous suspension were air dried on a glass slide and a KRS-5 plate for the X-ray and infrared spectroscopy investigations, respectively. A small drop of the diluted suspension was dried in air on a microgrid and a collodion film reinforced with carbon for electron microscopy and diffraction. The mount observed at low magnifications was shadowed with chromium. About 20 mg of the air-dried gel was used for simultaneous DTA (differential thermal analysis) and TG (thermogravimetry), which were carried out in nitrogen atmosphere and at a heating rate of 20°C per min. The infrared spectra were obtained from a disc prepared by mixing of 2 mg of the sample and 600 mg of KBr. An air-dried sample of the allophane, which was not preserved in water, and a diluted suspension prepared from it were analyzed in the same way as the gels.

OBSERVATIONS

Electron micrographs

An electron micrograph of the dispersed gel at low magnification (Fig. 1) shows a mat of threadlike units that are as much as a micrometer* long. In the micrograph at high magnification (Fig. 2), the threads are seen to consist of bundles or clusters of still-finer filiform units. Some of the finer threads, which commonly are in pairs, curve away from bundles causing them to appear frayed. This curving and pairing feature of the finer imogolite was noted in gel from pumice beds in Japan and was interpreted as an indication of the tubular habit of its structural unit (Wada *et al.*, 1970). The o.d. of the finer units is 18–20 Å, and the i.d. is approximately 7–10 Å. These diameters are similar to those of imogolite units in Japan. The Maui imogolite is much purer than that found previously at several places in Japan (Yoshinaga, 1968; Wada and Yoshinaga, 1969; Yoshinaga and Yamaguchi, 1970) but does contain a few granular

*The term micrometer (μm) was adopted for micron at the meeting of the International Committee on Weights and Measures, Paris, September 1968.

impurities (Figs. 1 and 2) similar to those found in the material from Japan. The impurities are probably allophane, but gibbsite may also be present because a small amount of this mineral was identified in the air-dried gel by DTA.

Electron micrographs of dispersed allophane at low and high magnification (Figs. 3 and 4) show it to occur mainly in fine granular particles and to be intimately intermixed with minor quantities of filiform imogolite. The imogolite threads occur in bundles or clusters (circled areas in Fig. 3) as in the gel, but they are considerably shorter. The allophane, as shown by these micrographs, has a peculiar porous texture, and most of it apparently occurs in subrounded particles having diameters of approximately 50 Å. These subrounded particles may be similar to those found in gels from several different sources (Yoshinaga, 1968; Wada and Yoshinaga, 1969; Yoshinaga and Yamaguchi, 1970) and allophane (Kitagawa, 1971) in Japan. Whether these particles are actually hollow spheres and are unit particles of allophane, as suggested by Kitagawa (1971), could not be verified. Isolated or clusters of ringlets (circled area in Fig. 4) are common in the allophane. Probably they represent cross-section of the tubular units of imogolite which stand up perpendicularly to the supporting film. Rather irregular arrangements of the tubular units of imogolite in the allophane and the superimposing of the texture of the supporting film on that of allophane do not permit definitive interpretation. However, the difference in the texture between allophane and imogolite seems to indicate that the change from one to the other involves more than a simple rearrangement of the chainlike molecules consisting of aluminum, silicon, and water or hydroxyls from the paracrystalline state or vice versa.

Diffraction data

Electron and X-ray diffraction patterns (Figs. 5 and 6) agree with those given for imogolites derived from pumice and volcanic ash (Yoshinaga *et al.*, 1968; Russell *et al.*, 1969; Wada and Yoshinaga, 1969) and confirm that the filiform material observed in the electron microscope is imogolite. The electron-diffraction pattern from randomly oriented threads (Fig. 5a) shows a series of rings among which the reflections at 1·40, 2·12, 2·3, 3·3, 3·72, 4·15, 5·7 and 7·8 Å can be seen on the print. Alignment of the threads results in strong arcing of some of these reflections (Fig. 5b), where the beam stopper lies nearly parallel to the fibre axis. The meridian reflections appear at 1·40, 2·12, and 4·15 Å, whereas the equatorial reflections appear at 5·7, 7·8 and 11·8 Å, the last of which is hardly seen on the print. The 2·3 and 3·3 Å reflections become
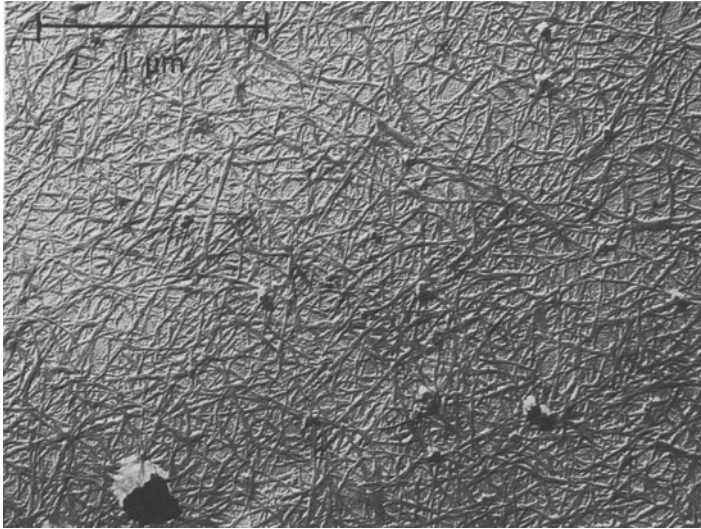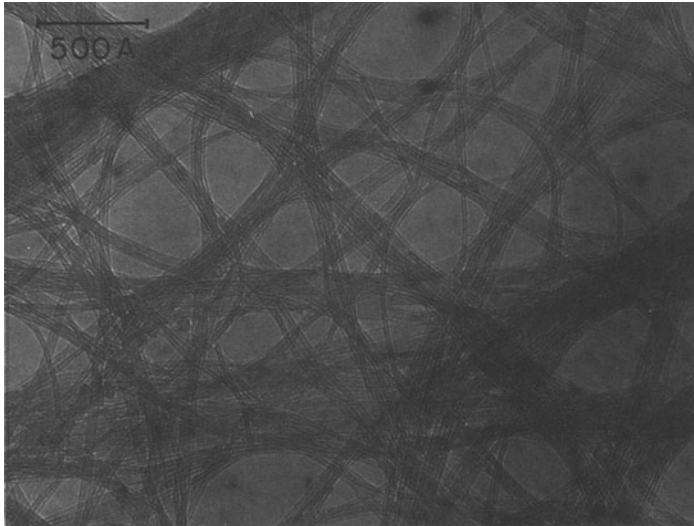
Fig. 1. Electron micrograph of gel, low magnification.

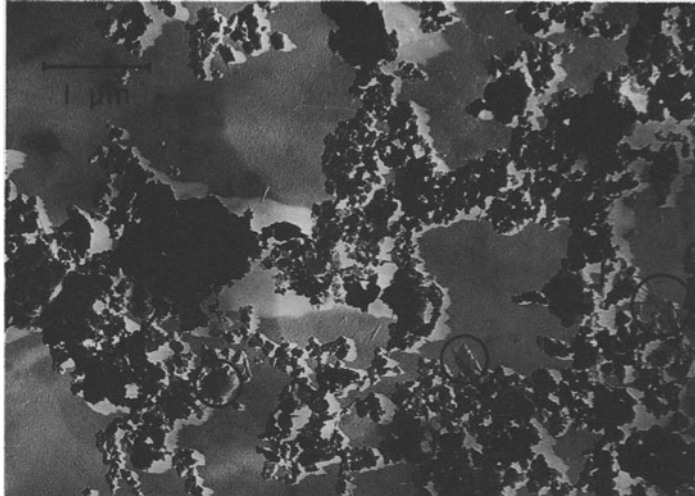Fig. 2. Electron micrograph of gel, high magnification.

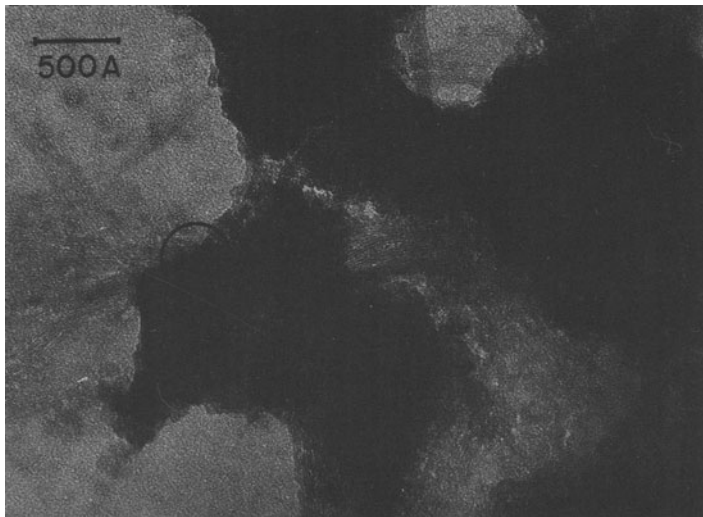Fig. 3. Electron micrograph of allophane, low magnification.

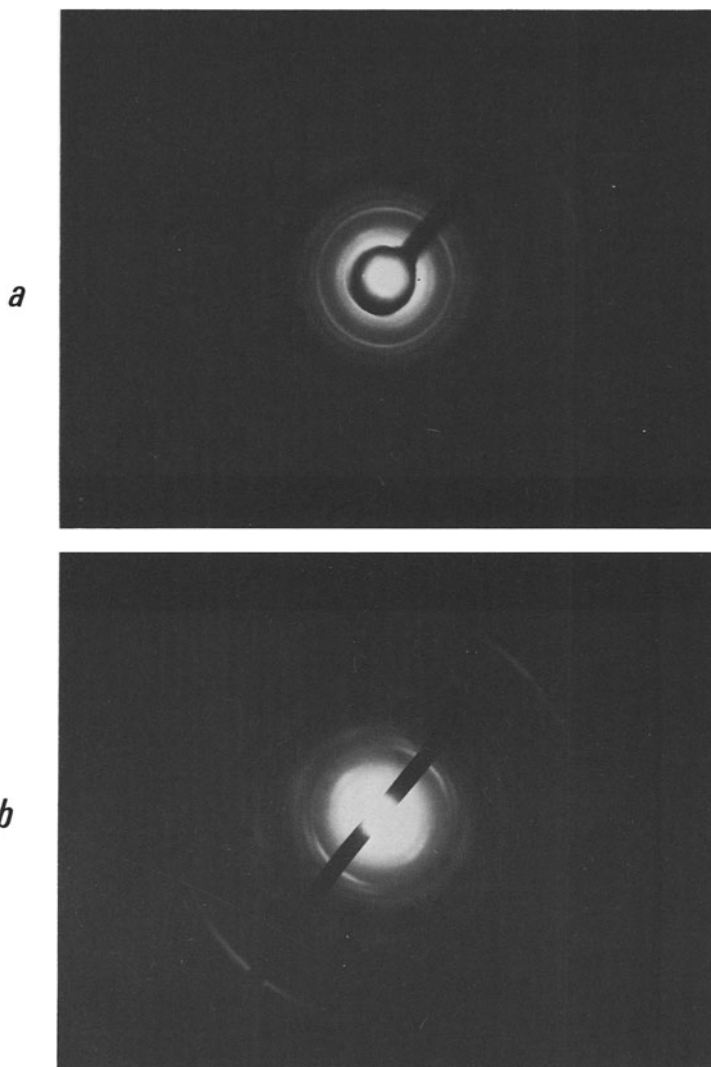Fig. 4. Electron micrograph of allophane, high magnification.

Fig. 5. Electron diffraction patterns of gel: (a) Imogolite threads in random orientation. (b) Imogolite threads in parallel orientation.
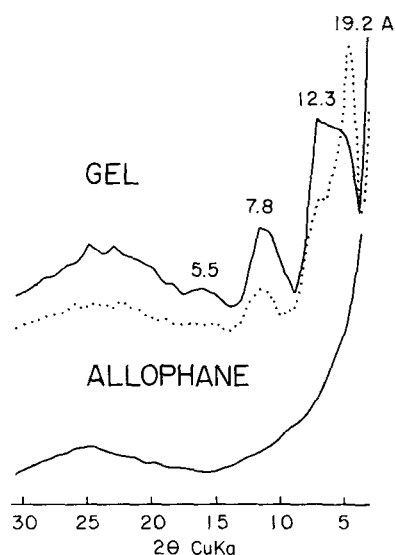
Fig. 6. X-ray diffraction patterns of gel and allophane. Dotted line, heated at 140°C and at one-half the intensity scale of other curves.

much broader but show discernible intensity maxima on the third layer line and equator, and the second and first layer lines, respectively. The 3·72 Å reflection also appears as a pair of arcs on the second layer line at an angle of 30° to the fiber axis. The sample examined by X-ray diffraction seems to be not very well oriented but showed the following characteristics of imogolite. Broad yet strong reflections occur at 12–19, 7·8 and 5·5 Å in the sample dried at room temperature; and the one at 18–19 Å increases remarkably upon heating at 140°C.

The X-ray diffraction pattern for the dispersed allophane (Fig. 6), unlike the one for the gel, has no discernible reflections, which supports the other evidence indicating that this material is noncrystalline. Two very broad rings with the spacings of about 2·3 and 3·3 Å appear in the electron diffraction pattern for the allophane. From a mass of filiform material in the allophane, such as shown in circled areas in Fig. 3, additional weak reflections appear at 1·40 and 4·15 Å, which coincide with strong meridian reflections of imogolite. The other reflections from imogolite, particularly the equatorial ones, are absent or much weaker than those from imogolite. Probably the virtual absence or weakness of these reflections arises from not only admixture of allophane but from an undeveloped state of imogolite threads.

Thermal-analyses data

Simultaneous DTA and TG curves of the air-

dried gel and allophane are shown in Fig. 7. The DTA curve for the air-dried gel shows three endothermic reactions with maxima at 135°, 280° and 390°C which are accompanied by the weight loss (TG curve) and an exothermic reaction at 975°C. The first endothermic reaction is due to removal of adsorbed water; the second and third are due to dehydroxylation of gibbsite and imogolite, respectively. The endothermic effect at 390°C was noted only as a very feeble reaction in the previous study (Patterson, 1964). Similar curves for the allophane show a more or less continuous dehydration, dehydroxylation, and weight loss with increasing heat. The pronounced exothermic reaction for the allophane and the weak one for the gel are the opposite of the strong reaction for imogolite and weaker one for allophane reported (Yoshinaga and Aomine, 1962; Miyauchi, 1966) for materials in volcanic ash and pumice in Japan. Part, if not all, of the exothermic reactions in both the gel and allophane are probably due to the formation of mullite (Patterson, 1964).
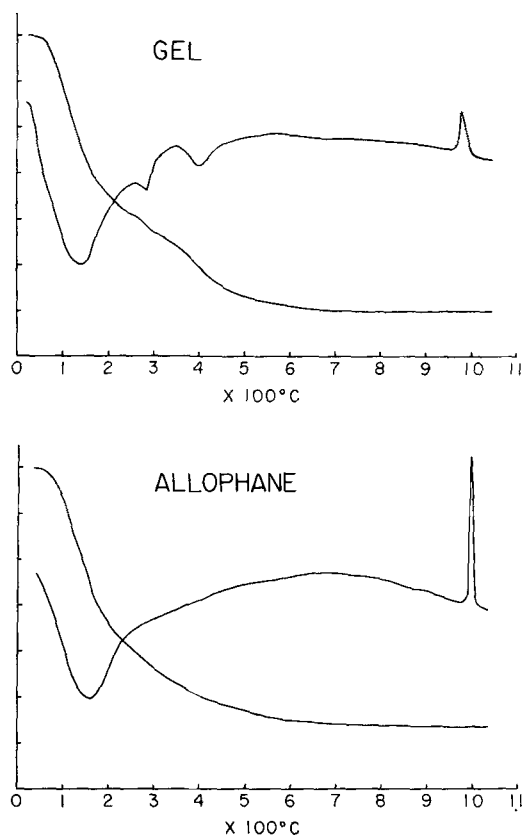

Fig. 7. DTA and TG curves of gel and allophane; scale of ordinate is 10 u.v. for DTA and 1 mg for TG curves.

Infrared Spectroscopy

The infrared spectrum of the dispersed gel (Fig. 8) shows two absorptions characteristic of imogolite at 995 and 935 cm^{-1} in the Si–O stretching region (Wada, 1966; Russell *et al.*, 1969). The poor resolution of these absorptions in the spectrum of the air-dried gel pressed in a KBr disc is due partly to the admixture of impurities and partly to random orientation of imogolite threads (Russell *et al.*, 1969). The purity of the dispersed gel indicated from the electron microscopy is further confirmed by comparison of its spectrum with those of the similarly dispersed gel from the Kitakami pumice bed. The Kitakami gel was one of the purest imogolites found in Japan (Wada and Yoshinaga, 1969), but the spectrum for it was the same as the one for the dispersed gel from Maui, only after the dithionite-citrate and 2 per cent Na_2CO_3 treatments. The untreated Kitakami gel showed stronger absorption, with maxima at 1100, 825, and 670 cm^{-1}.

The spectrum for the allophane (Fig. 8) shows absorption bands similar to those from the air-dried gel, but the spectrum for the dispersed allophane failed to show the resolution in the Si–O stretching region as shown in the dispersed gel. The similarities in the infrared spectra of the allophane and imogolite probably are caused by their similar chemical compositions, and differences are due to one being noncrystalline and the other consisting of molecules having an orderly arrangement.
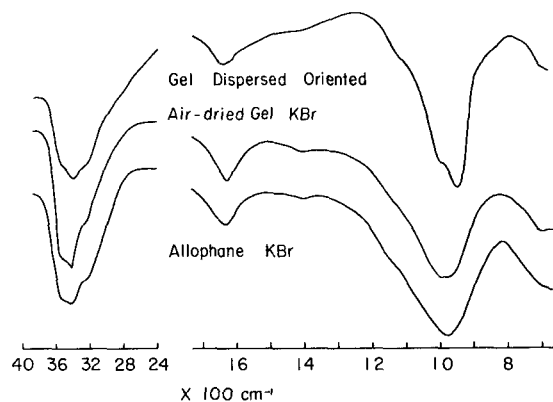

Fig. 8. Infrared spectra of gel and allophane.

GENETIC IMPLICATIONS

Since imogolite was first described by Yoshinaga and Aomine (1962), its occurrence in weathered volcanic ash has been reported in Japan (Aomine and Miyauchi, 1965; Kawasaki and Aomine, 1966; Kanno *et al.*, 1968; Wada and Tokashiki, 1972). Chile (Besoain, 1968/1969; Aomine *et al.*, 1972) and Papua (Greenland *et al.*, 1969), and in pumice tuff soils in West Germany (Jaritz, 1967). Rather pure occurrences of imogolite in gels in several pumice beds have also attracted attention of investigators (Miyauchi and Aomine, 1966; Yoshinaga, 1968; Yoshinaga and Yamaguchi, 1970; Tazaki, 1971). These reports indicate that imogolite is not an uncommon mineral, but heretofore it has been identified only in weathering products of pyroclastic materials. Sieffermann and Millot (1969) have described the occurrence of imogolite in Andosols derived from recent basaltic rocks in Cameroon, Central Africa. However, their identification of imogolite associated with allophane and layer silicates was made only on the basis of its fibrous habits observed in the electron microscope. Whether this imogolite formed by weathering of pyroclastic material or basaltic rocks is not clear from their description. The occurrence of imogolite in the gels derived from massive, though jointed, basalt rather than pyroclastic material on Maui, therefore, deserves attention and may provide the key to formation of imogolite.

The gels found in the basaltic saprolites in Maui and in the pumice beds in Japan are very much alike in their appearance and composition. They occur as fillings and coatings and coatings on vesicles, vugs, and cracks in the weathered rocks. This occurrence indicates that imogolite forms from dissolved silicate and aluminum or aluminate ions or from suspended hydrous silica and alumina derived from parent material. The dissolution or disintegration of the parent basalt during weathering operates with exceptional efficiency in Maui because of the following factors: (1) the exceptionally high rainfall in the vicinity of the sampling site, where the average annual rainfall is 250 in. according to the U.S. Weather Bureau maps; (2) the excellent subsurface drainage and permeability of the weathered material, a result of the soil and upper parts of the saprolite (above 20 ft) being so porous that ordinary rainfall is absorbed; and (3) the low pH and high organic content of the leaching solution. The water in similar saprolite collected from Kauai, Hawaii, ranged in pH from 4·0 to 5·9 (Patterson, 1971). The organic acids derived from decaying organic matter in the overlying soil under tropical rain forest contribute to the acidity and maintenance of aluminum in the leaching solution.

The related origin of imogolite and allophane is indicated by their close association in the weathered rocks and similar composition. Because the allophane has a sugary appearance and is covered by a clear or light-colored gel, it seems more logical to assume that the allophane forms from the gel than that the gel forms from the allophane. The results of these investigations, however, suggest that imogolite crystallizes from allophane, and the presence

of gibbsite in the gel and its virtual absence in the allophane seem to support this theory. Crystallization of imogolite and gibbsite probably took place as desilication progressed in the weathered rocks. The association of the gel and gibbsite has also been noted in the weathered pumice bed in Japan (Wada and Matsubara, 1968; Yoshinaga and Yamaguchi, 1970; Tazaiki, 1970; Aomine and Mizota, 1972). Whether this association means that imogolite is an intermediate product in the transformation of allophane to gibbsite or that these materials form separately was not determined. Field and microscopic observations in Japan indicate that gibbsite forms separately, possibly because of the heterogeneity of parent materials (Wada and Matsubara, 1968).

The observation and theories outlined in the foregoing paragraph are those of the first three authors and are based on laboratory work on the Maui gel and allophane and considerable field and laboratory work on weathered pyroclastic rocks and soil in Japan. Patterson agrees that their excellent work disproves his earlier conclusion of the allophane having formed from the gel (Patterson, 1964), but the field evidence against the reverse order of formation is difficult to ignore. The occurrence of the gel and allophane clearly indicates that the gel and allophane were introduced by shallow-percolating water, and the process active at the time of sampling was causing accumulation rather than dissolution and removal. A change from the solid allophane to the very hydrous gel, if it took place, would most likely have taken place during leaching, and under such conditions, masses of gel probably would not have formed. Also, the overlapping of the gel and its extent beyond and above the allophane is opposed to the formation of imogolite from allophane. Furthermore, laminae in the gel seem much more likely to result from periodicity in deposition controlled by climatic cycles than from cycles of alteration.

Though the gel and allophane are clearly related in origin, perhaps the conclusion that one forms from the other is incorrect. The position of the allophane between the host saprolite and the gel indicates that part of it is older, and possibly these materials formed in slightly different chemical environments. Differences in age and variations in the organic-matter content of the water from which the gel and allophane formed might have caused the following characteristics of these materials: (1) the abundant laminae in the gel and the absence of layering in the allophane; (2) superposition of the gel on the allophane; (3) the absence of evidence for alteration of an intermediate stage between imogolite threads and the granular allophane, as shown by the electron micrographs. If the allo-

phane is considerably older, it may have been introduced before this area became heavily forested, and, therefore, the transporting water may have contained less organic material and was less acid than it is now.

The confirmation of imogolite in the Maui gel and the additional information on the allophane make it seem likely that these materials are widely distributed through the weathered rocks in Hawaii. The materials described herein are the only ones sufficiently pure for study found during fieldwork on ferruginous bauxite deposits. However, several authors cited in the introduction of this article and Patterson (1971) have reviewed reasons for believing that noncrystalline materials occur widely. Much of this material is probably allophane, and some of it formerly thought to lack crystallinity may be imogolite.

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Résumé – Un gel minéral et une allophane recueillis dans une saprolite basaltique de Maui, Hawaii, et étudiés par Patterson en 1964, ont fait l'objet d'un nouvel examen. Le constituant principal du gel est l'imogolite, et la gibbsite et l'allophane en sont des constituants mineurs. Les diagrammes de diffraction électronique et de rayons X, la courbe d'ATD et le spectre infrarouge du gel sont caractéristiques de l'imogolite. L'allophane est virtuellement amorphe aux rayons X, mais elle contient une petite quantité d'imogolite en aiguilles relativement courtes. Les micrographies électroniques à haute résolution indiquent des différences dans l'organisation structurale de l'allophane et de l'imogolite et suggèrent que l'imogolite a cristallisé à partir de l'allophane.

Kurzreferat – Anorganisches Gel und Allophan, erhalten aus basaltischem Saprolit auf Maui, Hawaii, und studiert von Patterson im Jahre 1964, wurde neu untersucht. Der Hauptbestandteil des Gels ist Imogolit, und die untergeordneten Bestandteile sind Gibbsit und Allophan. Die Elektron- und Röntgenbeugungsmuster, DTA Kurve und ein Ultrarotspektrum des Gels sind kennzeichnend für Imogolit. Das Allophan ist praktisch unkristallin gegenüber Röntgenstrahlen, erthält jedoch eine kleine Menge von Imogolit in verhältnismässig kurzen Fasern. Elektronmikrographien mit hoher Auflösung zeigen Unterschiede in der Gefügeanordnung zwischen Allophan und Imogolit und deuten auf mögliche Kristallisation von Imogolit aus Allophan.

Резюме — Повторно рассматривались неорганический гель и аллофан, найденных на базальтовых сапропелитах в Мави, Гавайи, которые исследовались в 1964 г. Паттерсоном. Главный компонент геля — имоголит, а гиббсит и аллофан являются второстепенными компонентами. Электронограмма; дифракционная картина; кривая дифференциального термического анализа и инфракрасный спектр геля характерны для имоголита. По рентгенограмме видно, что аллофан почти что некристаллической структуры, но содержит малое количество волокнистного имоголита. Электронные микроснимки показывают разницу структурной организации аллофана и имоголита и наводят на мысль, что имоголит образовался от кристаллизации аллофана.