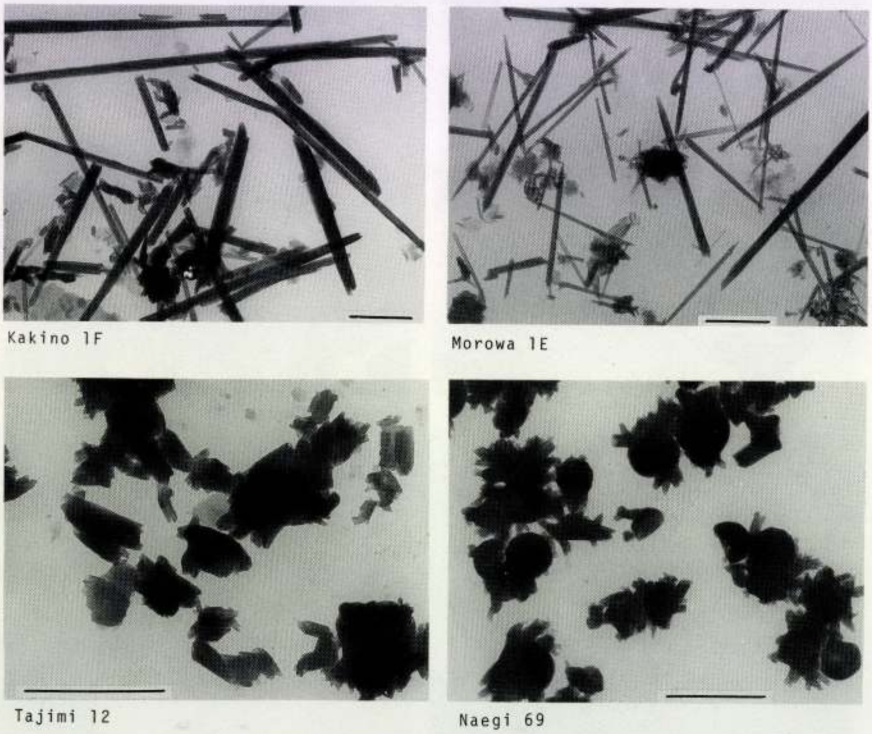
TABLE 2. Mineralogical properties

Sample	$(H_{10}/A)_{56}$*	b†	Morphology*	A_{3700}/A_{3620}†
Halloysite				
Kakino 1F		8.921	L	1.088
Morowa 1E		8.928	L	1.221
Tajimi 12		8.930	SBP	1.512
Naegi 69	0.20	8.915	BT	1.500
Naegi 72	0.50	8.908	BT	1.050
Imaichi	1.23	8.912	B	0.725
Iki 1	0.38	8.941	B	1.110
Naegi 60	0.30	8.925	B	1.262
Komaki 116A	0.35	8.942	S	1.208
Komaki 118B	0.57	8.920	S	1.128
Mure 1	0.46	8.923	BT	0.891
Chitose	0.47	8.941	B	0.798
Yame 9	0.78	8.919	B	0.969

*See text.

†For samples dried at 110°C for 2 hours.



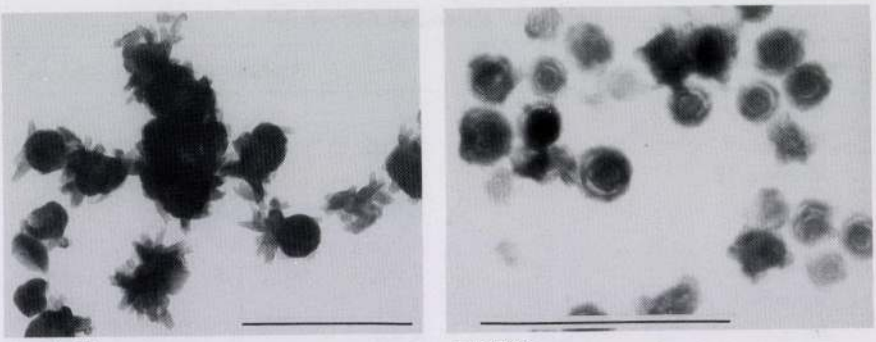
Kakino 1F

Morowa 1E

Tajimi 12

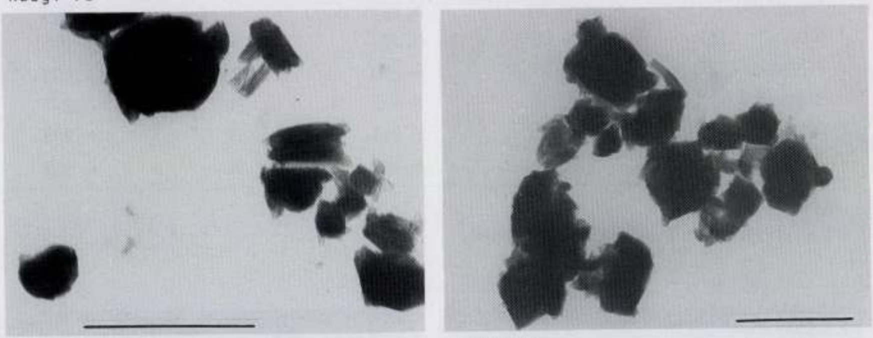
Naegi 69

FIG. 1. Electron micrographs. Bar scale – 1 μm.



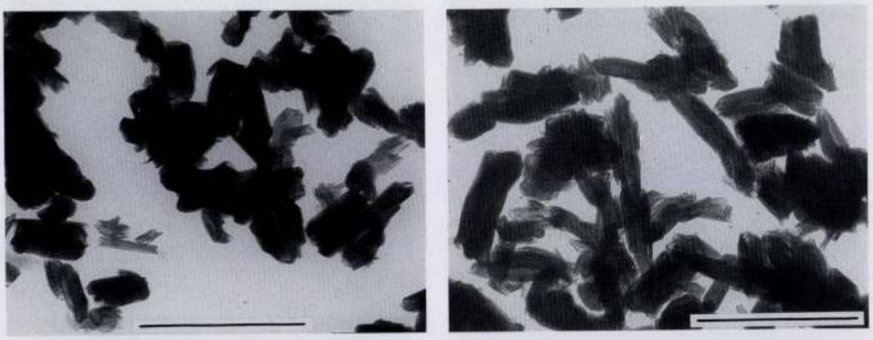
Naegi 72

Imaichi



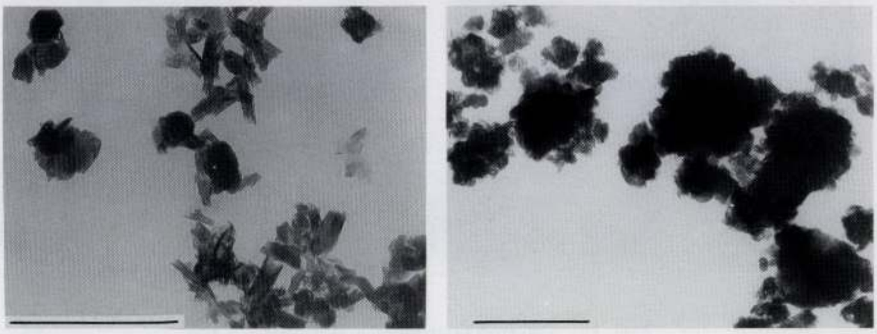
Iki 1

Naegi 60



Komaki 116A

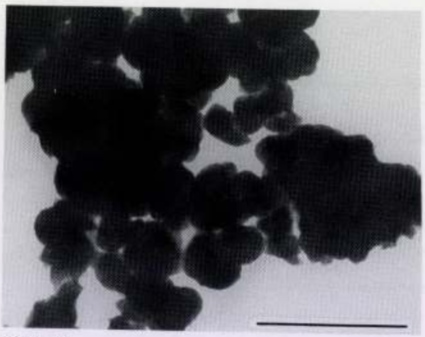
Komaki 118B



Mure 1

Chitose

FIG. 1. (continued)



(continued)

110°C for 2 hours, and they, as well as kaolinite, were put into silica-glass tubes of 4 mm in inner diameter to the depth of about 28 mm. Instrumental conditions were the same throughout.

Determination of Fe content was carried out both before and after removal of Fe oxides. The Fe removal was done by reducing samples with sodium dithionite powder in 0.3 M sodium citrate – 1 M $NaHCO_3$ solution (Jackson, 1956). Fe was determined by atomic absorption for samples decomposed with HCl – HF.

RESULTS AND DISCUSSION

ESR spectra of 13 halloysite samples and 4 samples of other minerals are shown in Figs. 2 and 3, respectively. These halloysite spectra are similar to those of kaolinite which are characterized by signals near $g = 4$ and near $g = 2$. The signals for kaolinite near $g = 4$ are attributed to Fe^{3+} at the octahedral sites (Jones *et al.*, 1974; Meads and Malden, 1975). It holds true also for halloysite; Table 3 shows that halloysite with large $g = 4$ signals contains a large amount of structural Fe, that is Fe after the dithionite treatment. The line shape of $g = 4$ signals depends on the distortion of the crystalline field around Fe^{3+}. In the case of kaolinite, the line shape varies from sample to sample and may be related to the degree of stacking disorder (Jones *et al.*, 1974; Meads and Malden, 1975). The present results indicate that long tubular halloysite shows complex group of signals near $g = 4$ in contrast to spheroidal halloysite with a single signal. This fact implies that the long tubular halloysite has more ordered structure. The signals for kaolinite near $g = 2$ are attributed mainly to lattice defects probably related to substitution of Mg for Al (Angel *et al.*, 1974). Some halloysites show signals in this region. Tajimi 12 which shows distinct signals here contains a considerable amount of kaolinite.

In addition to the signals mentioned above, some halloysites show a very broad signal around $g = 2$. Imaichi, Iki 1, Chitose, and Yame 9 are examples. This signal may be due to spin interaction between closely situated Fe^{3+} ions, and therefore is attributed to contamination with Fe minerals, for example hydrous Fe sesquioxides. The spectrum for goethite in Fig. 3 confirms this inference. Thus, ESR is useful as a fast and reliable way to distinguish structural Fe from Fe contamination.