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MÖSSBAUER SPECTRA OF SOIL KAOLINS FROM SOUTH-WESTERN AUSTRALIA

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Abstract—Six well-characterized soil kaolins from widely separated sites in south-western Australia and four reference kaolins were studied by ^{57}Fe Mössbauer spectroscopy at room temperature after removal of non-structurally-bound iron with dithionite-citrate-bicarbonate solution. The soil kaolins and one of the reference kaolins were also studied at temperatures near 16 K. The soil kaolins were remarkably similar in crystal size, crystallinity, dehydroxylation temperature, cation exchange capacity, surface area and iron content. Mössbauer spectra of the soil kaolins at room temperature were also essentially identical consisting of a quadrupole-split doublet superimposed on a broad component which indicated that all of the iron was present as Fe(III) and that slow paramagnetic relaxation effects were present. Mean values for the chemical isomer shift and quadrupole splitting of the doublet for the soil kaolins were 0.33 and 0.55 mm/s respectively which indicates that the iron is in the octahedral sites of the kaolin lattice. The spectra of the soil kaolin samples at temperatures near 16 K showed a further slowing down of the paramagnetic relaxation and confirmed that no discrete iron oxide minerals were present.

Mössbauer spectra of the four reference kaolins at room temperature showed a doublet component similar to those for the soil kaolins. Three of them showed evidence for other spectral components including, in two cases, a component due to the presence of Fe(II).

Key Words—Iron, Kaolin, Mössbauer spectroscopy.

INTRODUCTION

Kaolin is the most abundant and widespread clay mineral in deeply weathered soils in south-western Australia (Singh, 1991). The crystallinity of soil kaolin varies considerably but kaolins from highly weathered soils of tropical and sub-tropical regions are usually disordered and of sub-micron size (Hughes and Brown, 1979; Dixon, 1989; Singh and Gilkes, 1992). The presence of iron in the kaolin structure contributes towards the poor crystallinity of the mineral (Herbillon *et al.*, 1976; Mestdagh *et al.*, 1980). Several techniques including bulk chemical analysis, infrared spectroscopy, electron paramagnetic resonance, Mössbauer spectroscopy, electron microprobe analysis, and single crystal analytical electron microscopy indicate the presence of iron in the kaolin structure (Dixon, 1989; Singh and Gilkes, 1992). Since soil kaolin generally occurs in a mixture of minerals, some of which contain iron, it is difficult to assign the iron to particular minerals on the basis of the above techniques. Mössbauer spectroscopy has been successfully used to determine the presence of iron in kaolin in the absence of other iron-containing minerals (Malden and Meads, 1967; Fysh *et al.*, 1983; Murad and Wagner, 1991). It is therefore likely to prove of value in the investigation of forms of iron in purified soil kaolin especially since the signal produced is specific to the iron in a sample.

The aim of this study of soil kaolins was to determine: (1) whether iron was within the kaolin structure or in other minerals, e.g., iron oxides; (2) the oxidation state of the iron; (3) whether structurally bound iron was in the octahedral or tetrahedral sites of kaolin. The results of Mössbauer spectroscopy are also compared with Fe contents determined by bulk chemical analysis using X-ray fluorescence (XRF) and single crystal analysis using analytical electron microscopy (AEM).

MATERIALS

Soil kaolins

The six soil samples were collected from within an area of about 100,000 km^2 in south-western Australia. The soils of the region are mostly derived from deeply weathered Precambrian crystalline rocks (mainly granite and gneiss with intrusion of doleritic dykes). The deeply weathered profiles have been reworked by colluvial and alluvial processes giving a complex pattern of soils ranging from deeply weathered lateritic profiles in upland situations to colluvial and alluvial modifications of these soils on slopes and valley floors. Soil patterns vary systematically with topography and climatic factors. Six representative and almost pure soil kaolins were selected for the present study on the basis of a much more extensive study of the mineralogy of west Australian soils (Singh, 1991). Complete description and other details of the samples are published elsewhere (Singh and Gilkes, 1992).

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Table 1. Some properties of soil and reference kaolins.

Sample no.	001 reflection d-spacing (Å)	001 reflection WHH (°2θ)	HB index	Dehyd. temp. (°C)	CEC cmol(+) kg^{-1}	N_2-BET surface area (m^2/g)	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	TiO_2 (%)	K_2O (%)	AEM Fe_2O_3 (%)
124	7.195	0.36	5.8	488	5.23	51	42.45	35.83	2.16	1.11	0.29	2.7
150	7.286	0.48	5.5	496	4.88	51	43.43	36.17	2.48	1.82	0.16	2.3
167	7.211	0.44	5.6	492	5.03	52	42.60	35.57	2.43	1.29	0.34	2.1
169	7.211	0.36	6.1	488	4.23	53	43.08	36.07	2.53	1.21	0.16	2.3
175	7.193	0.33	6.2	488	5.38	56	43.40	36.90	2.23	1.04	0.15	2.6
235	7.205	0.38	5.0	488	4.42	44	43.54	35.47	2.81	1.10	0.25	2.8
239	7.193	0.44	4.8	484	4.75	55	43.01	34.15	3.34	0.98	0.35	2.8
Georgia	7.199	0.27	43.7	540	0.42	24	46.11	36.73	0.42	1.84	0.05	0.3
Wungong	7.184	0.26	11.6	514	2.44	nd	45.35	38.40	0.41	0.91	0.17	nd
Greenbushes	7.167	0.27	15.9	535	[1]3.60	[1]24	46.00	39.20	0.19	0.01	0.14	nd
Goomalling	7.173	0.40	11.2	530	[1]3.50	[1]28	44.80	37.10	0.75	1.80	0.53	nd

WHH = width at half height; HB = Hughes and Brown (1979) crystallinity index; CEC = cation exchange capacity; nd = not determined; [1] = values taken from Bolland *et al.* (1976); AEM = analytical transmission electron microscopy.

Free iron oxides were removed from the soil kaolins using a sequence of three extractions with dithionite-citrate-bicarbonate (DCB) solution (Mehra and Jackson, 1960). The properties of the kaolins were determined using XRD, thermal, and electron-optical methods as described by Singh and Gilkes (1992). There was no evidence for the presence of free iron oxides in the DCB treated kaolins.

Reference kaolins

(i) Georgia kaolin. Sample MP#5 was obtained from Georgia Kaolin Company, U.S.A. It contains kaolinite and small (<5%) amounts of mica and anatase.

(ii) Wungong kaolin. This sample is a laterite pallid zone clay over a mafic rock and consists of kaolinite and halloysite. It occurs near Wungong Dam, south-western Australia.

(iii) Greenbushes kaolin. Laterite pallid zone clay over pegamatite from Greenbushes, south-western Australia. A mineral kaolinite deposit.

(iv) Goomalling kaolin. Laterite pallid zone clay over granite from Goomalling, south-western Australia. A mineral kaolinite deposit.

METHODS

Mössbauer spectroscopy

Mössbauer spectra were recorded in constant acceleration mode with a symmetric double ramp waveform and were subsequently folded to eliminate the parabolic background. A 20mCi ^{57}Co in rhodium source was used. The velocity scale was calibrated with reference to the spectrum of an α-iron foil at room temperature, the centre of the six line pattern being taken as zero velocity. For low temperature measurements, the sample was mounted in a closed cycle helium gas expansion refrigerator (Air Products Expander Module DE 202).

About 300 mg of kaolin was spread evenly in a 25 mm diameter holder for room temperature measurements and in a 19 mm diameter holder for low temperature measurements. The folded spectra consist of 250 channels with about 2 × 10^6 and 10 × 10^6 counts per channel for the soil and reference kaolins respectively at room temperature. For the low temperature spectra, data were collected with about 17 × 10^6 counts per channel for the soil kaolins and 40 × 10^6 counts per channel for the Georgia kaolin.

Spectral analysis

Each Mössbauer spectrum was fitted with singlets, doublets, and sextets of Lorentzian absorption peaks using a sum of squares minimization routine. During the fitting procedure the areas and widths of each line of the doublet components were constrained to be equal. For the sextet components the area ratio of the outer to middle to inner pair of lines was constrained to be 3:2:1. Values of the chemical isomer shift, δ; the quadrupole interaction, Δ; magnetic hyperfine field splitting, B_{hf}; linewidth, Γ; and spectral area of each component were allowed to vary freely during the fitting procedure unless otherwise stated.

RESULTS AND DISCUSSION

General properties of the kaolins

Various properties of the soil and reference kaolins are given in Table 1. Minor amounts (<5%) of mica were identified by XRD in all the reference kaolins; however, in the soil kaolins no other clay mineral was observed. All the soil kaolins were b-axis disordered (Bailey, 1980). The Wungong kaolin contained 0.7 μm tubular halloysite crystals along with platy kaolinite crystals and the remaining reference kaolins were quite well ordered, platy kaolinites. The accurately measured 001 spacing for the soil kaolins (except sample #150) did not differ significantly from the value for the reference kaolins indicating that no substantial interstratification with other clay minerals occurred. Dehydrox-

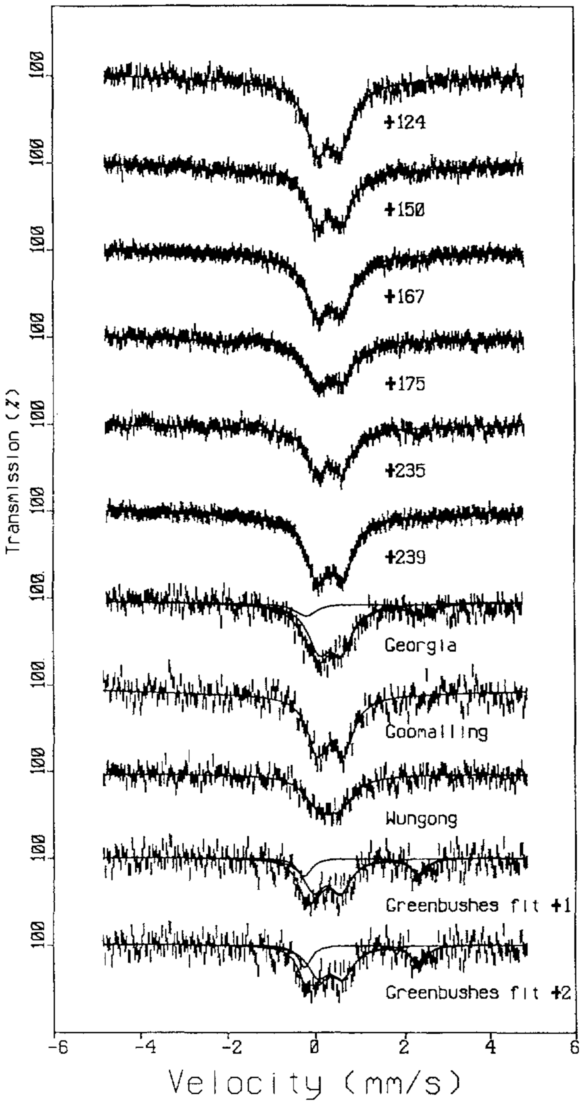

Figure 1. Mössbauer spectra of kaolin samples at room temperature.

ylation temperature and Hughes and Brown crystallinity index (Hughes and Brown, 1979) were significantly lower and width at half height (WHH) of basal reflections and surface area were significantly higher for the soil kaolins compared to standard kaolins. The increase in WHH is mostly caused by the smaller crystal size which is consistent with the higher values of surface area and the electron microscopy data for the soil kaolins (Singh and Gilkes, 1992). Cation exchange capacity (CEC) for the soil kaolins (mean = 4.85 cmol(+) kg^{-1}) was higher than for the reference kaolins. Georgia kaolin had a much lower CEC than the three kaolins from laterite pallid zone. There was no systematic change in 001 spacing with increasing iron content but observations for a larger number of soil kaolins (n = 15) indicate that the 060 spacing does show a tendency to increase with iron content (Singh, 1991).

Table 2. Parameters of the Mössbauer spectral doublets for the kaolins at room temperature.

Sample	δ (mm/s)	Δ (mm/s)	Γ (mm/s)	%A
#124	0.33	0.53	0.60	—
#150	0.34	0.55	0.54	—
#167	0.33	0.55	0.61	—
#175	0.32	0.55	0.64	—
#235	0.33	0.56	0.54	—
#239	0.33	0.58	0.59	—
Georgia				
Fe(III)	0.34	0.50	0.60	77
Fe(II)	1.12	2.63	0.60	10
Goomalling	0.34	0.58	0.55	—
Wungong[1]	0.30	0.47	0.74	—
Greenbushes (Fit #1, $\chi^2 = 0.833$)[1]				
Fe(III)	0.26	0.60	0.55	70
Fe(II)	1.03	2.57	0.39	30
Greenbushes (Fit #2, $\chi^2 = 0.848$)[1]				
Fe(III)	0.33[2]	0.55[2]	0.58	66
Fe(II)	1.04	2.56	0.37	34

δ is the chemical isomer shift relative to the centre of an α-iron foil spectrum at room temperature, Δ is the quadrupole splitting, Γ is the full linewidth at half height, and %A is the percentage spectral area of each component. The error in δ and Γ is about ±0.02 mm/s and the error in Δ is about ±0.03 mm/s.

[1] These spectra showed very little or none of the broad component that is due to paramagnetic relaxation.

[2] For this fit these parameters were constrained to be the mean values of the parameters for the soil kaolins.

The most significant difference in chemical composition between the soil and reference kaolins is the presence of greater amounts of iron in the soil kaolins as determined by both XRF analysis of bulk clay and analysis of single crystals using analytical transmission electron microscopy.

In summary it is clear from the data in Table 1 that all six soil kaolins are smaller in crystal size, have poorly ordered structures, and contain greater amounts of iron compared to the reference kaolins. Also of significance is the remarkable similarity in the properties of the soil kaolins despite their origins in quite separate areas of south-western Australia.

Mössbauer spectral properties of the kaolins

Mössbauer spectra of the soil kaolins at room temperature are shown in Figure 1. The spectra are all similar and consist of a broad linewidth doublet (with maximum absorption of about 1.3%) superimposed on a very broad component that gives the spectra the appearance of having a curved background that dips down towards the centre of the spectrum. The spectra were computer fitted with a doublet and a broad singlet. The spectral parameters of the doublet are given in Table 2.

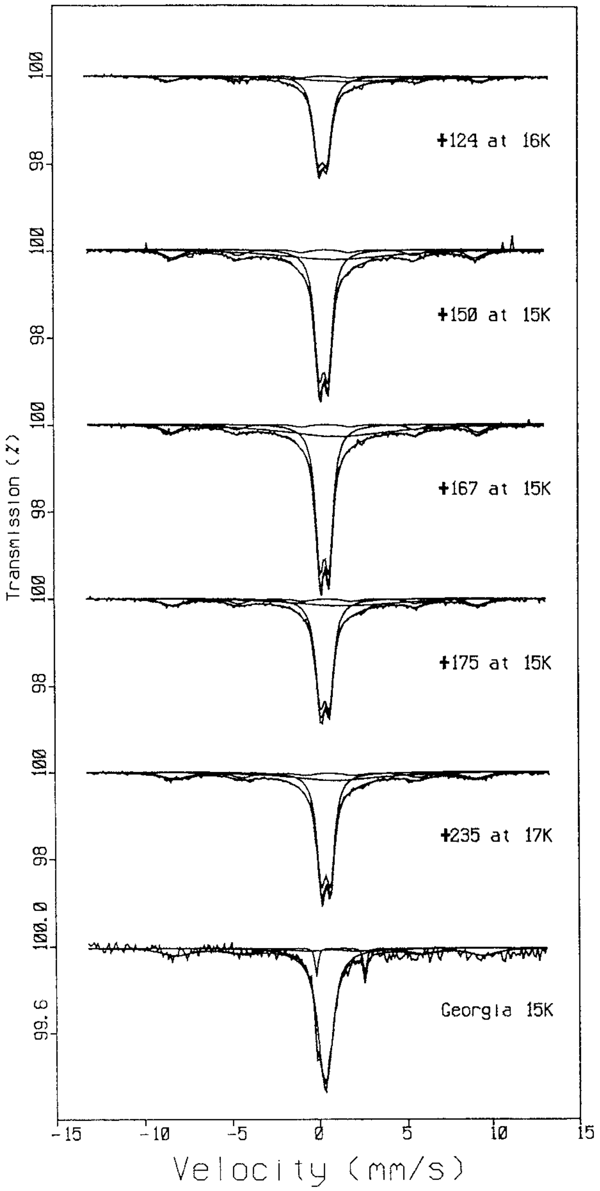

Figure 2. Mössbauer spectra of kaolin samples at temperatures near 16 K.

The values of δ and Δ for the doublets indicate that the oxidation state of the iron is Fe(III) and that it is mostly octahedrally coordinated (Malden and Meads, 1967). Attempts to fit doublets with the parameters of Fe in the tetrahedral sites of kaolin, as given by Petit and Decarreau (1990), resulted in the areas of the tetrahedral doublets being reduced to less than 3% of the area of the octahedral doublet. Thus, with the given signal to noise ratio, it appears that more than about 95% of the Fe atoms are in octahedral sites. The spectral parameters of the doublet and the presence of the very broad component are consistent with previous Mössbauer spectral observations of natural kaolins (Murad and Wagner, 1991; Fysh *et al.*, 1983). The very broad spectral component can be attributed to slow paramagnetic relaxational effects of iron within the kaolin structure. Such effects can only be expected if the iron atoms are well separated. These well separated iron atoms must be within the kaolin host structure rather than exchangeable iron on the kaolin surface both because the clays have been treated for the removal of iron oxides and because they were Na^+ saturated. The low concentration of iron (<3.5%) in these samples is also consistent with this observation. There is no evidence for the presence of Fe(II) in these samples.

The similarity of the spectral parameters of the doublet with those associated with some small particle iron oxide minerals at room temperature means that it is difficult to ascertain whether small particle iron oxide phases are still present. However, low temperature Mössbauer measurements can distinguish between iron structurally bound within kaolin and iron in microcrystalline iron oxides (Murad and Wagner, 1991). Murad and Wagner used this technique to show that treatment of kaolins with dithionite removed goethite.

The Mössbauer spectra of some of the soil kaolins at about 16 K are shown in Figure 2. The spectra consist of a central doublet, a broad component due to paramagnetic relaxational effects (similar to those in the room temperature spectra) and a sextet of peaks. The spectral parameters from the computer fitting procedure are given in Table 3 and are consistent with those reported for dithionite treated kaolins (Murad and Wagner, 1991). The value of the magnetic hyperfine field splitting of the sextet component (Table 3) indicates that it is due to paramagnetic iron that is relaxing very slowly rather than to iron in an iron oxide which would give a lower magnetic hyperfine field splitting and narrower resonant lines. Attempts to fit the spectra with two sextets corresponding to slowly relaxing paramagnetic iron and iron in an oxide resulted in the area of the iron oxide sextet being reduced to zero. Constraining the area of the iron oxide sextet to be 5% of the total spectral area resulted in increased values of χ^2. This indicates that less than about 5% of the iron is in the form of oxides. The paramagnetic sextet component in the spectra indicates iron that is in the kaolin lattice in a dilute form so that spin-spin relaxational effects are minimal. The iron giving rise to the doublet component probably has closer iron neighbours which would cause faster paramagnetic relaxation via spin-spin interactions. The broad component is due to iron with intermediate relaxation times of the order of the Mössbauer measurement time (about 10^{-8} s).

Thus the Mössbauer spectra of the soil kaolins at low temperatures indicate that they are essentially free from iron oxides.

Table 3. Mössbauer spectral parameters for the kaolins at temperatures near 16 K.

Sample		δ (mm/s)	Δ (mm/s)	B_{hf} (T)	Γ (mm/s)	%A
#124 at 16 K	doublet	0.39	0.52	—	0.66	57
	sextet	0.54	−0.04	55.3	1.30	16
#150 at 15 K	doublet	0.41	0.53	—	0.61	51
	sextet	0.47	−0.05	53.8	1.26	17
#167 at 15 K	doublet	0.41	0.53	—	0.59	52
	sextet	0.48	−0.02	54.7	1.34	14
#175 at 15 K	doublet	0.40	0.54	—	0.63	55
	sextet	0.48	−0.08	54.2	1.40	19
#235 at 17 K	doublet	0.41	0.54	—	0.61	52
	sextet	0.52	−0.20	53.4	1.90	22
Georgia at 15 K	Fe(III) doublet	0.32	0.35	—	0.84	66
	Fe(II) doublet	1.21	2.77	—	0.20	6
	sextet	0.64	−0.07	54.7	2.00	28

δ is the chemical isomer shift relative to the centre of an α-iron foil spectrum at room temperature, Δ is the quadrupole splitting, B_{hf} is the magnetic hyperfine field splitting, Γ is the full linewidth at half height (for the outer lines in the case of a sextet), and %A is the percentage spectral area of each component. The error in δ and Γ is about ±0.02 mm/s, the error in Δ is about ±0.03 mm/s, and the error in B_{hf} is about ±0.5T.

Reference kaolins

The Mössbauer spectra of the four reference kaolins (i.e., Georgia, Goomalling, Wungong, and Greenbushes) were also measured at room temperature (Figure 1). The spectral parameters for these kaolins are given in Table 2. All four of these spectra were of lower intensity (about 0.2% maximum absorption) than those from the soil kaolins which is consistent with the lower iron content of these samples as determined by bulk chemical analysis and single crystal AEM (Table 1).

Georgia kaolin. The spectrum is fitted with two doublets, corresponding to octahedral Fe(III) and an Fe(II) component, and a broad component corresponding to paramagnetically relaxing iron. The spectral parameters are given in Table 2. If it is assumed that the recoil free fractions (or Mössbauer effect efficiencies) of the different forms of iron are approximately the same at room temperature, then the spectrum indicates that about 10% of the iron is in the form of Fe(II). Fe(II) has previously been observed in kaolin (Murad and Wagner, 1991; Malden and Meads, 1967) and has been doped into synthetic kaolinites (Cuttler, 1980). The spectral parameters of the Fe(II) component in the Georgia kaolin are very close to those measured in the previous studies. Some mica is present in this sample and may also contain some structural Fe.

The Mössbauer spectrum of this sample at 15 K was also recorded (Figure 2). The spectrum is fitted with a sextet corresponding to slowly relaxing paramagnetic iron, a doublet corresponding to Fe(II), and an unresolved doublet corresponding to Fe(III). The parameters derived from the fit are shown in Table 3. The possible presence of forms of iron other than iron in the kaolin structure may account for the unresolved Fe(III) doublet. Small (less than 1% of the total spectral area) sextet components corresponding to iron oxides could be fitted to the spectrum but the poor signal to noise ratio of the sextet makes it difficult to confirm the presence of magnetically ordered iron oxides.

Goomalling kaolin. The Mössbauer spectrum of this sample gives parameters that are similar to those of the soil kaolins (Table 2). Thus iron is probably in the same structural sites as in the soil kaolins but is present at a lower concentration.

Wungong kaolin. Fitting the spectrum with a doublet gives parameters that are somewhat different from those of the soil kaolins (Table 2). This is because the doublet is not resolved in this spectrum. In addition, the broad component due to the slowly relaxing paramagnetic iron is either absent or very small. The spectral shape is most probably a result of the presence of more than one form of iron, e.g., small particle iron oxide in addition to iron bound within the kaolin structure.

Greenbushes kaolin. The Mössbauer spectrum of this sample consists of two doublets but again the broad background component is absent or very small. Table 2 shows the spectral parameters of the doublets derived from two computer fits to the spectral data. In fit #2 one of the doublets has its parameters constrained to be those of the average of the soil kaolin doublets. The two doublets indicate the presence of both Fe(III) and Fe(II). If it is assumed that the recoil free fractions of the two forms of iron are approximately the same at room temperature, then the spectra indicate that about 30% of the iron is in the form of Fe(II). The spectral parameters of the Fe(II) component are similar to those for the Georgia kaolin and other published values for

Fe(II) in kaolin (Murad and Wagner, 1991; Malden and Meads, 1967; Cuttler, 1980).

GENERAL DISCUSSION

The data presented here are consistent with published Mössbauer spectral observations on Fe(III) in kaolin (Fysh *et al.,* 1983; Murad and Wagner, 1991). However, no soil kaolins contained Fe(II) which has been reported to occur in some other kaolins. The Mössbauer spectral parameters of the soil kaolins indicate that the iron is solely in the form of Fe(III). The similarity of the parameters with those of iron in gibbsite suggests that the iron in the kaolinite is in the octahedral cation sites of kaolin (Malden and Meads, 1967). The presence of the slowly relaxing paramagnetic component in the spectra indicates that the iron must be in a dilute form in the kaolin lattice thus minimising spin-spin relaxation effects.

The higher iron content and lower degree of structural order of the soil kaolins compared to the reference kaolins is in agreement with previous observations that there is an association between iron content and poor structural order in kaolins.

The Fe(II) in Greenbushes kaolin has spectral parameters that are very close to those reported previously for some kaolins. It is unlikely to be associated with an impurity since the parameters are significantly different from those for muscovite which was the only Fe-bearing impurity detected. Less than 2% muscovite can be present as the K_2O content of the clay is 0.14% (Table 1). Much greater amounts (about 10% muscovite) would be required to generate the observed Fe(II) spectral intensity. Pyroxenes, amphiboles, biotite and ilmenite occur in the parent pegmatite and associated rocks. These minerals contain Fe(II) but do not persist during lateritic deep weathering and were not detected by XRD which has a detection limit of about 1%. We therefore propose that the Fe(II) in the Greenbushes kaolin occupies octahedral sites in the kaolin structure.

Perhaps the most remarkable finding of this research is the great similarity of the six soil kaolins with respect to both their Mössbauer spectra and all other measured properties. This may indicate that the particular properties of these kaolins represent a stable, equilibrium form of kaolin for the soil conditions existing in south-western Australia. Furthermore, it can not be argued that these kaolins have been simply inherited from erosion of the deep kaolinitic laterite pallid zones that mantle the landscape of south-western Australia. The properties of pallid zone kaolins (Wungong, Greenbushes, Goomalling) are distinctly different from those of the soil kaolins so that if they were the source of soil kaolins they must have experienced some pedogenic alteration.

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